EPA 910-R-01-003

United States
Environmental Protection
Agency

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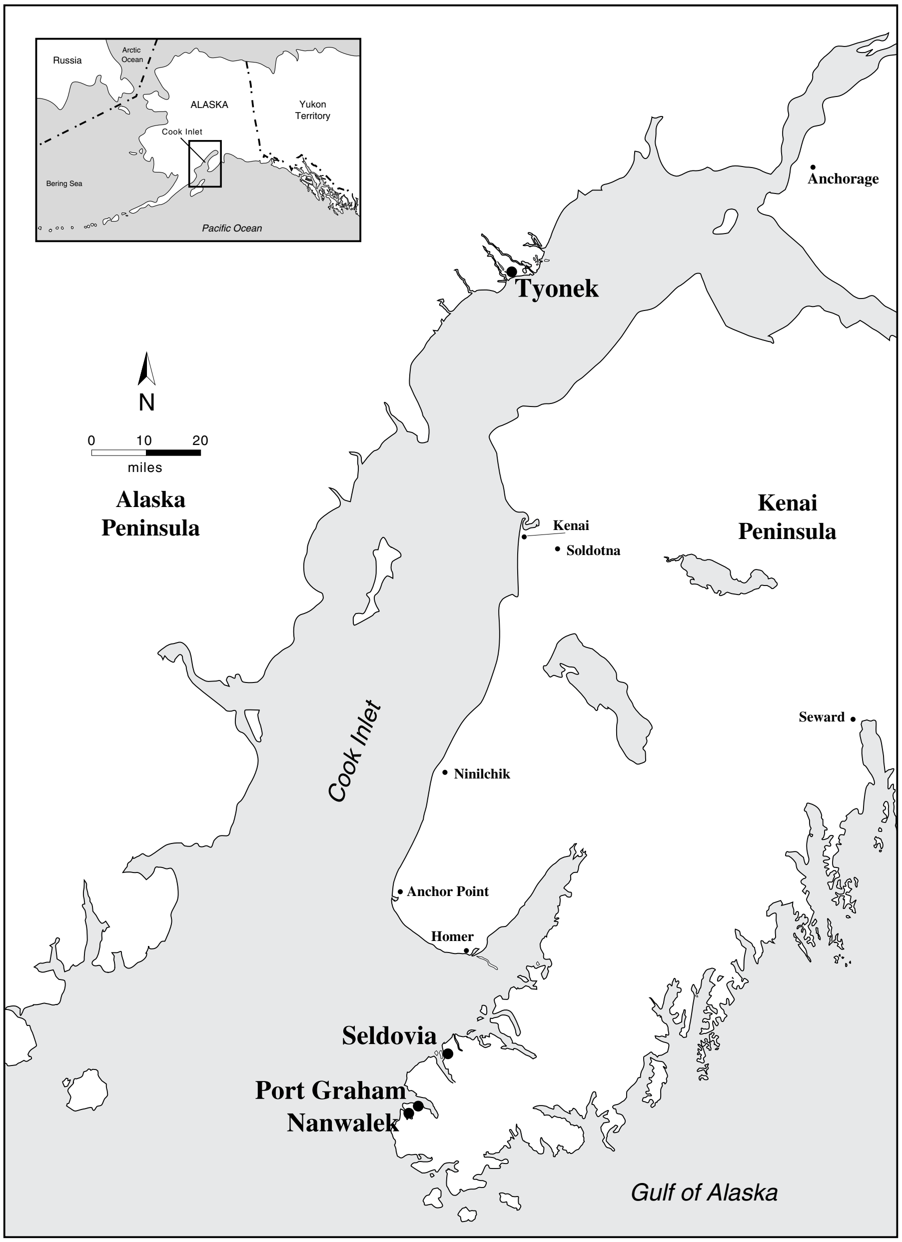
Office of Environmental Assessment

December 2003

Appendix G

QAPP for Samples and

QAPP for sample homogenization, compositing & analysis



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Quality Assurance Project Plan
for the
Cook Inlet Contaminant Study Sampling

June 1997

Prepared by

United States Environmental Protection Agency
Office of water
Off:-- of Science and Technology

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References

1. USEPA. "EPA Requirements for Quality Assurance Plans for Environmental Operations." Draft Interim Final. August 1994. EPA QA/R-5

1. Introduction and Scope (Group A)

The purpose of this document is to present the methods and procedures that will be used for the collection of environmental samples in the Cook Inlet region of Alaska for analytical testing and the quality assurance procedures that will be employed. This document addresses only the sample collection effort.

As stated by the USEPA$_{(1)}$, a Quality Assurance Project Plan (QAPP) is an essential document for environmental data operations. Its purpose is to define in detail how specific Quality Assurance (QA) and Quality Control (QC) activities will be implemented during a project. This QAPP was prepared according to guidance presented in the document USEPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, USEPA QA/R-5, Draft Interim Final, August 1994. Reference to the QAPP elements described in the USEPA document are included herein. The sample collection methods, procedures and protocols shall follow, where practical, the guidelines as recommended in the document titled *Guidance For Assessing Chemical Contaminant Data For Use in Fish Advisories. Volume I: Fish Sampling and Analysis, Second Edition* (EPA 823-R-95-007).

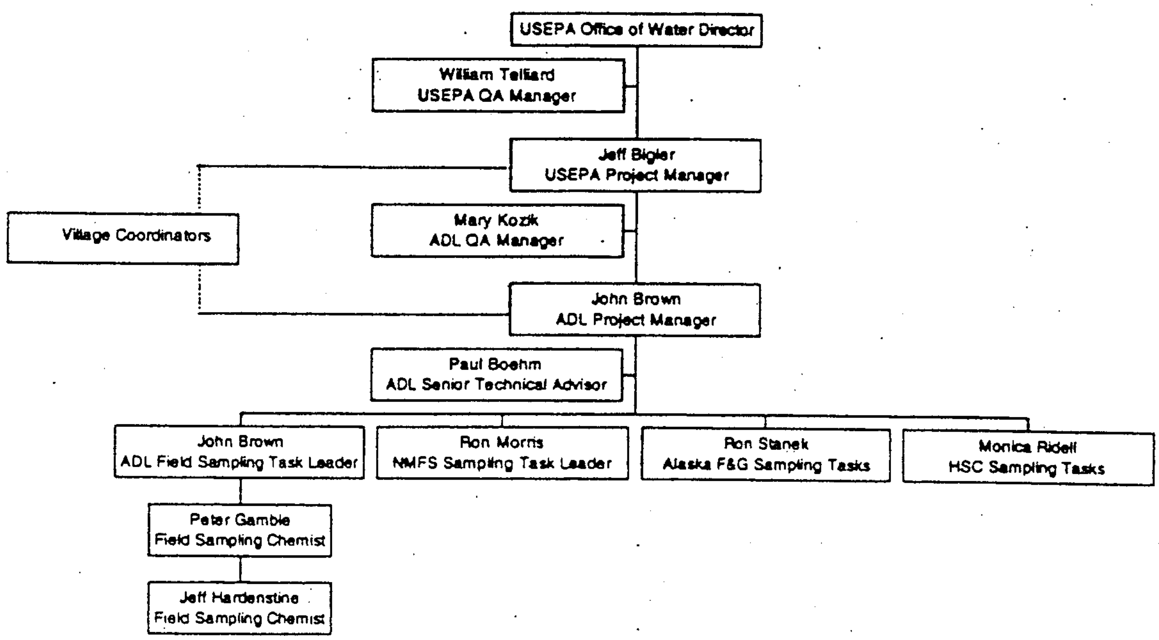
1.1 Project Organization (A4)

This section identifies the individuals and organizations participating in the project and discusses their specific roles and responsibilities.

1.1.1 Project Organization Diagram

The lines of authority, reporting and communication for this project are shown on Figure 1. Specific responsibilities are discussed below.

Figure 1: Project Organization Structure



1.1.2 Roles and Responsibilities

USEPA Project Manager

The USEPA Project Manager is Mr. Jeff Bigler. The USEPA Project Manager has responsibility for USEPA oversight of the activities and for approval of the workplan and QAPP.

USEPA Quality Assurance Manager

The USEPA Quality Assurance Manager is Dr. William Telliard. The USEPA Quality Assurance Manager has the responsibility to review and approve all Quality Assurance Project Plans (QAPPs). Additional responsibilities for the project include:

- Reviewing and evaluating field procedures
- Conducting external performance and system audits of the procedures

Arthur D. Little Project Manager

The Arthur D. Little Project Manager is Mr. John Brown. The Arthur D. Little Project Manager is responsible for implementing the project and has the authority to commit the resources necessary to meet project objectives and requirements. The Arthur D. Little Project Manager's primary function is to ensure that technical, financial and scheduling objectives are achieved successfully. The Arthur D. Little Project Manager reports directly to the USEPA Project Manager and will provide the major point of contact and control for matters concerning the project.

The Arthur D. Little Project Manager will:

- Define project objectives and develop a detailed work plan schedule
- Establish project policy and procedures to address the specific needs of the project
- Acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule constraints
- Communicate with the project team concerning the project objectives
- Monitor and direct the Task Leaders
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness
- Review and analyze overall task performance with respect to planned requirements and authorizations
- Approve all reports (deliverables) before their submission to USEPA
- Ultimately be responsible for the preparation and quality of all reports
- Represent the project team at meetings and public hearings

Arthur D. Little Quality Assurance Manager

The Arthur D. Little Quality Assurance Manager for this project is Ms. Mary Kozik. The Arthur D. Little QA Manager is responsible for ensuring that all procedures for this project are being followed. The Arthur D. Little QA Manager will provide assistance to the USEPA and Arthur D. Little Project Managers in the preparation of the workplans and QAPP and their distribution.

Additional specific functions and duties include:

- Reviewing and approving QA plans and procedures
- Providing QA technical assistance to project staff
- Reporting on the adequacy, status, and effectiveness of the QA program

Field Responsibilities

Field Sampling Task Leader

The Field Sampling Task Leaders are responsible for leading and coordinating the day to day activities of the various resource specialists under their supervision.

Specific responsibilities include:

- Coordination with the USEPA and Arthur D. Little Project Manager on technical issues in specific areas of expertise
- Implementing field related activities with assurance of schedule compliance and adherence to project management requirements

- Coordinating and managing technical field staff
- Implementing specified QC procedures
- Adhering to work schedules provided by the USEPA and Arthur D. Little Project Manager
- Authoring reports of field activities
- Identifying problems at the task level, resolving difficulties in consultation with the USEPA and Arthur D. Little Project Manager, and implementing and documenting corrective action procedures
- Maintaining communication between the field task and the project management
- Participating in preparation of the final report.

The Field Sampling Task Leaders and a brief description of their task follows.

ADL Field Sampling Task Leader

Mr. John Brown, Arthur D. Little
Arthur D. Little will be responsible for the collection of field samples in the Villages.

NMFS Field Sampling Task Leader

Mr. Ron Morris, National Marine Fisheries Service
The NMFS Field Sampling Team will be responsible for the collection of Beluga Whale samples and coordinating with Arthur D. Little and USEPA.

ADF&G Field Sampling Task Leader

Mr. Ron Stanek, State of Alaska, Department of Fish and Game
The ADF&G Field Sampling Team will be responsible for the collection of Salmon samples at the Tyonek Village; coordinating with the HSC Field Sampling Team for the collection of Seal samples, and coordinating the Tyonek and seal sampling with Arthur D. Little and USEPA.

HSC Field Sampling Task Leader

Ms. Monica Ridell, Harbor Seal Commission
The HSC Field Sampling Team will be responsible for collection of Seal samples in coordination with the ADF&G Field Sampling Team, Arthur D. Little, and USEPA.

Field Sampling Technical Staff

The Field Sampling Technical Staff report directly and work under the supervision of the Field Sampling Task Leader. The field sampling team will be comprised of scientific staff with specialization and technical competence in field sampling activities to effectively and efficiently perform the required work. Responsibilities include:

- Performing all work in adherence with the workplan, QAPP, and relevant SOPs
- Notifying immediately the Arthur D. Little Field Sampling Task Leader of any problems encountered outside normal operating procedures
- Completing fully all required documentation

Field Sampling Technical Staff are responsible for the maintenance of sample custody and appropriate documentation. Custody procedures are required to ensure the integrity of the samples with respect to prevention of contamination and maintenance of proper sample identification during handling. In this role, they are responsible for:

- Receiving and inspecting the sample containers
- Verifying chain-of-custody documentation and its correctness
- Signing appropriate documents
- Assigning tracking numbers to each sample and maintaining documentation
- Initiating transfer of the samples to appropriate destinations.
- Controlling and monitoring access to samples while in their custody

Other Roles and Responsibilities

Arthur D. Little Senior Technical Advisor

Dr. Paul Boehm will act as advisor to the Arthur D. Little Project Manager to assist in resolution of technical issues and to aid in the implementation of the field sampling task.

Village Coordinators

Village Coordinators are responsible for assisting the USEPA and Arthur D. Little Project Manager in the selection of sample types and the timing of sample collection. The Village Coordinators are selected by the USEPA in consultation with Village Leaders. The Village Coordinators are:

Tyonek	Robert Stephan
Seldovia	Lillian Elvsaas
Port Graham	Walter Megannek
Nanwalek	Nancy Yeaton and Tom Evans

1.2 Project Background (A5)

Under the direction of the USEPA Office of Science and Technology, Engineering and Analysis Division (EAD), dietary food items samples are being collected from the Cook Inlet region of Alaska to support the development of a human health risk assessment of Native American tribes living in coastal Alaska.

Under this program, EAD has funded an Interagency Agreement (IA) with the Minerals Management Service (MMS) to conduct a field program to collect samples of marine organisms, including fish, marine invertebrates, tissue from marine animals, and marine plants.

The MMS is currently conducting an environmental study of a portion of the Cook Inlet region of Alaska and has contracted the services of Arthur D. Little to conduct a number of project related tasks. The IA was developed to utilize the MMS contractor to improve efficiency and economy.

1.3 Project Description (A6)

1.3.1 Summary

In consultation with the USEPA, Arthur D. Little will coordinate with the Alaskan Villages of Tyonek, Port Graham, Nanwalek, and Seldovia to identify locations within the Alaska designated Subsistence Harvest Areas where samples of specific dietary food items are routinely harvested by the Village members.

In consultation with the USEPA Project Manager and the EPA-designated Village Coordinators, Arthur D. Little will coordinate the collection of whole samples of finfish, marine invertebrates, marine mammal organs, and marine plants from these identified areas during the summer of 1997. Arthur D. Little shall follow field collection protocols and methods as identified and/or approved by the USEPA Project Manager. Arthur D. Little will then ship all of the samples to a laboratory identified by the USEPA Project Manager.

All work for this project will be conducted under the requirements specified in the Quality Assurance Project Plan (QAPP) provided by Arthur D. Little and approved by the USEPA Project Manager.

Arthur D. Little will provide a final comprehensive report of all activities related to this project.

1.3.2 Project Schedule

Quality Assurance Project Plan Preparation (Task 1)

Arthur D. Little will develop a detailed Quality Assurance Project Plan (QAPP) for this project. This plan will describe the quality assurance requirements for the project and the system for ensuring that they are met. The QAPP will be developed following the requirements described in EPA QA/R-5. Upon completion, the QAPP will be submitted to the USEPA Project Manager for review and approval. This task will be completed prior to the arrival of the ADF&G or ADL Field Sampling Team in the Villages.

Identification of Sampling Locations (Task 2)

The Arthur D. Little Project Manager, in consultation with the USEPA Project Manager and USEPA -designated Village Coordinators, will identify the most appropriate locations for collecting each of the samples identified by USEPA (e.g., salmon, bivalves, resident fish, plants, octopus, seal and whale) from each of the four villages, as suitable. The sample locations must be those same locations where the Native Alaskans typically harvest the specified dietary food items.

This task will be completed prior to the field sampling with modification to location details throughout the project as required.

Planning of Field Sampling (Task 3)

The Arthur D. Little Project Manager, in consultation with the USEPA Project Manager, will coordinate the timing for collection of the samples with the four Village Coordinators and others identified by or agreed to by the EPA Project Manager. Any and all contact between the Arthur D. Little Project Manager and ADL Field Sampling Team and State, Federal, and private organizations regarding any aspects of this study will be made through the USEPA Project Manager.

The ADL Field Sampling Team, in consultation with the USEPA Project Office and ADF&G, will procure from the Village harvesters all of the samples identified by USEPA (not to exceed a total of 100 composite samples). Procurement will be conducted in a manner as agreed to with each of the Village leaders, ADF&G, and the USEPA Project Manager.

This task will be completed prior to the field sampling with modification to species collected and number individuals throughout the project as required.

Field Sampling (Task 4)

During a 12-day period and no later than September 1997, the ADL Field Sampling Team will collect, prepare for shipment by composite group, and ship all collected samples to a designated location using sampling methods and protocols consistent with the US EPA guidance document titled Guidance For Assessing Chemical Contaminant Data For Use in Fish Advisories. Volume I: Fish Sampling and Analysis.

This task is currently scheduled to begin on June 15, 1997 and end on June 27, 1997.

The ADL Field Sampling Team will work with and assist the Village members and others in the collection of the samples to ensure that all of the samples are obtained (not to exceed a total of 100 composite samples). The Arthur D. Little Project Manager shall assign one field staff member to each of the three villages of Seldovia, Port Graham, and Nanwalek for the purposes of working with the Village Coordinators, fishermen, and gatherers in order to ensure collection of all samples.

In addition, in consultation with the USEPA Project Manager, the Arthur D. Little Project Manager will oversee additional sampling tasks.

- Collection of the Harbor Seal samples is coordinated with and performed by ADF&G, The Harbor Seal Commission, the Cook Inlet Marine Mammal Council, and the Village Coordinators;
- Collection of the Beluga samples is coordinated with and performed by ADF&G and NMFS and the Village Coordinators;
- Collection of the Tyonek salmon species is coordinated with and performed by ADF&G and the Tyonek Village Coordinator;

- Collection of all other samples is coordinated with the Village Coordinators and performed by Arthur D. Little personnel.

Arthur D. Little shall contact the USEPA Project Manager every other day during the field collection effort for the purpose of providing a status report. The USEPA Project Manager will be notified immediately of problems related to the successful completion of field efforts. Changes (locations, species, and numbers of samples and individuals per composite) may be made to the sampling plan as agreed to by the USEPA Project Manager and the Village Coordinators.

Reporting (Task 5)

Within 4 months of completion of the field collection effort, Arthur D. Little will submit to the USEPA Project Manager a final comprehensive report detailing the field collection effort, including details pertaining to all aspects of the work performed by the ADL Field Sampling Team and the other field sampling teams. The report will also include specific locations of all sampling efforts in the form of latitude/longitude data and the relationship between the sampling locations and the State-designated subsistence areas.

Arthur D. Little shall provide one draft to the USEPA Project Manager and allow one month for comments. Arthur D. Little shall incorporate USEPA Project Manager comments into the final draft of the document.

1.4 Quality Objectives and Criteria (A7)

1.4.1 Objectives

This project is primarily a sample collection effort, and as such quality objectives are related to sample handling issues. Discussion of conventional data quality measures is included in this section.

The overall objectives of the project are:

- Collect samples representative of subsistence foods
- Collect representative samples of the identified foods
- Minimize contamination of sampling during collection, handling, and shipment

The project plan includes procedures designed to address these specific objectives.

To ensure that species representative of subsistence food are collected, the USEPA Project Manager and the Arthur D. Little Project Manager will work with the Village Leaders and Village Coordinators to properly identify sample types.

To ensure that representative samples of the identified foods are collected, the sampling effort will be conducted during optimal tide conditions during a peak harvest period. Also. individual samples will be composited prior to analysis to increase the representativeness of the sample set.

To minimize contamination of samples during collection, specific procedures will be followed. These include the use of gloves for sample handling, clean glass jars, clean foil and polyethylene bags for sample packaging, and adherence to operating procedures for each collection effort.

1.4.2 Criteria

This section describes the project objectives in terms of precision, accuracy, representativeness, completeness, and comparability

Precision
Precision is a measure of the degree to which two or more measurements are in agreement. As the analytical testing is beyond the scope of this project, no specific criteria are required for this parameter. However, sufficient sample volumes will be collected to allow for the assessment of precision during testing.

Accuracy
Accuracy is the degree of agreement between an observed value and an accepted reference value. Again, as analytical testing is beyond the scope of this project, no accuracy criteria are set. However, proper sample handling procedures to minimize sample contamination will be followed. Also, assessment of contamination, through the analysis of trip blank samples will be implemented. These steps are included to aid in the assessment of accuracy.

Completeness
Completeness, in the case of this project, is the number of valid samples collected relative to the number of samples that are planned to be collected. The completeness goal for this project is 90%.

It should be noted that sample types, locations, and numbers may change over the course of the sampling tasks based on local conditions and the availability of the selected species. This must be considered when assessing completeness. In brief, the goal is achieved when all of the available samples are collected and shipped with no errors in documentation or sample handling procedures.

Representativeness
Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter, variations at a sampling point, a process condition, or an environmental condition. Representativeness of the target organisms for this study was established by discussions between the village coordinators the USEPA Project Manager.

The representative goal will be satisfied by ensuring that once the sample types and locations are identified, it is those samples that are collected.

Comparability

Comparability is an expression of the confidence with which one data set can be compared with another. Comparability is dependent upon the proper design of the sampling program and the adherence to generally accepted sampling techniques, standard operating procedures, and this QAPP.

1.5 Project Narrative (A8)

The project is described in narrative form in Section 1.2 and 1.3.

1.6 Special Training Requirements or Certifications (A9)

All Field Sampling Staff will be experienced in environmental activities involving the collection of biota. They will also be familiar with operation of fishing equipment and small boats.

The Field Sampling Task Leaders are responsible for ensuring the village gatherers are properly trained for collection of the different samples.

1.7 Documentation and Records (A10)

1.7.1 Field Record

Whenever a sample is collected, a detailed description of the sample will be recorded on a Field Record Form (Figures 2, 3, and 4). This record will document the sample type, location, and other sample details.

The actual samples are assigned tracking numbers which are also recorded on the form to identify the sample to the record. The samples are identified by use of sample tags with sample numbers, sampling locations, and date and time of collection.

1.7.2 Field Notes

Field notebooks will provide the means of recording field data collection activities. As such, entries will be described in as much detail as possible so that a particular event or situation could be recreated without reliance on memory.

Field notebooks will be bound survey books or notebooks and will be assigned to field sampling staff.. The title page of each notebook will contain the following:

- person to whom the notebook is assigned
- notebook number
- project name
- project task start and end dates

Entries into the notebook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered.

Notes regarding the samples collection activities will be recorded. All entries will be made in ink, signed or initialed and dated and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark which is signed or initialed and dated by the sampler.

A vital notebook entry is the location where the samples are collected. The longitude and latitude of the sampling location will be identified using a current NOAA charts which are accurate to approximately 0.1 nautical miles.

The equipment used to collect samples will be noted, as well as other pertinent sampling details including the time of sampling, sample description, depth at which the sample was collected, volume and number of sample containers.

1.7.3 Final Report Records

The final report will include narrative description of the field activities. Copies of field records will also be included in the report. Also included will be a summarized listing of samples identifications, sample types, and sampling locations.

1.7.4 Record Retention and Final Disposition

Original field records and field notebooks are to be sent to the Arthur D. Little Project Manager at the completion of each individual task. Copies of these records should be maintained by the originator for reference should problems occur that require resolution.

Copies of all shipping logs, airbills, and other related documentation will also be forwarded to the Arthur D. Little Project Manager.

All original documents will be provided to USEPA within 6 months of submission of the final report.

Specification for retention of field samples by the receiving location are outside the scope of this document. While in storage, it is recommended that unused samples be stored with the original labeling materials.

2. Measurement and Data Acquisition (Group B)

2.1 Sampling Design (B1)

2.1.1 Approach

The sampling approach is designed to collect samples of marine organisms representative of the historic and routine harvest and consumption by the represented Native Alaskan tribes. This is the primary objective of the project. The types of samples were selected because they are commonly consumed in the area and are of high commercial, recreational, and subsistence fishing value based on interviews with Village members. The number of each type of sample and the compositing scheme was set by USEPA based on experience with other similar studies.

For practical reasons, the sampling design also considered species that are relatively abundant, easy to capture or collect, and large enough to provide sufficient tissue for chemical analysis. The samples collected are intended to be representative of those used by the Alaska Villages. As such, only legal harvestable sizes (as per subsistence guidelines) will be collected.

The collection volumes are designed considering the following compositing requirements:

- samples should be of similar size with the smallest length or weight no less than 75% of the largest
- samples are collected at the same time
- sufficient numbers are collected to provide adequate tissue for analysis

2.1.2 Sampling Plan

General

Each fish sample composite is comprised of five individual samples, with the possible exception of halibut where the number of individual samples may be reduced depending upon the size of the available catch.

Mammal samples will be comprised of one individual.

Octopus samples will be comprised of individual organisms; not a composite.

Plant and invertebrate composites will be comprised of a **minimum** of five individuals, but enough individuals to total approximately 300 grams of tissue sample.

All sampling sites will be located within designated subsistence harvest areas

Sample Locations and Numbers

The following details the samples that will be collected in each area:

Tyonek Village

6 Whole Fish Composite Samples comprised of:

1 Chinook Salmon x 3 composites

1 Sockeye Salmon x 3 composites

6 Marine Mammal Organ Samples comprised of:

1 Beluga x 3 organs
1 Seal x 3 organs

Seldovia Village
7 Whole Fish Composite Samples comprised of:
1 Red Salmon x 3 composites
1 Chinook Salmon x 1 composites
2 resident species (e.g., Bullhead, Halibut) x 3 composites

15 Whole Invertebrate Composite Samples comprised of:
1 Clam species (e.g., Butter Clam) x 3 composites
1 chiton species x 3 composites
1 mussel species x 3 composites
1 snail species x 3 composites
1 clam species from Ninilchik area (e.g., Razor Clam) x 3 composites

Port Graham Village
11 Whole Fish Composite Samples comprised of:
1 Chum Salmon x 3 composites
1 Chinook Salmon x 2 composites
2 resident species (e.g., Flounder, Seabass) x 3 composites

12 Whole Invertebrate Composite Samples comprised of:
1 clam species (e.g., Butter Clam) x 3 composites
1 chiton species x 3 composites
1 octopus species x 3 individuals
1 snail species x 3 composites

6 Marine Plant Composite Samples comprised of:
1 kelp species x 3 composites
1 Goosetoungue Plant x 3 composites
(only edible portion of the plant samples are collected)

Nanwalek Village
9 Whole Fish Composite Samples comprised of:
1 Sockeye Salmon x 3 composites
2 resident species (e.g., Cod, Halibut) x 3 composites

12 Whole Invertebrate Composite Samples comprised of:
1 Clam species (e.g., Butter Clam) x 3 composites
1 chiton species x 3 composites
1 octopus species x 3 individuals
1 snail species x 3 composites

3 Marine Mammal Organ Samples comprised of:
1 Harbor Seal x 3 organs

6 Marine Plant Composite Samples comprised of:
1 kelp species x 3 composites
1 Goosetongue Plant x 3 composites
(only edible portion of the plant samples are collected)

2.2 Sampling Methods Requirements (B2)

This section describes the procedures for collecting samples.

2.2.1 Sample Integrity

General

A critical requirement of the project is the maintenance of sample integrity from the time of collection to the shipment and arrival at the final destination. Sample integrity is preserved by preventing the loss of contaminants that might be present in the sample and the prevention of the introduction of contaminants during handling.

The loss of contaminants can be prevented in the field by ensuring that the sample collected is intact. Sample collection procedures should be performed with the intention of minimizing the laceration of fish skin and the breakage of invertebrate shells or carpaces. Once collected, integrity is maintained through careful and controlled sample handling and storage and preservation procedures.

Sources of extraneous contamination can include contamination from the sampling gear, oils and greases on ships, spilled fuel, skin contact, contact with soil or sand, exhaust, as well as a number of other unanticipated sources. All potential sources should be identified before the onset and during sample collection, and appropriate measures should be taken to minimize or eliminate them. The following are some examples:

- Boats should be positioned so that engine exhaust does not fall on the deck area where samples are being collected
- Ice chests and other sample storage containers should be scrubbed clean with detergent and rinsed with distilled water prior to use
- Samples should not be placed directly on melting ice, but should be stored inside plastic bags first
- Proper gloves should be used when handling samples and gloves should be discarded after each use

Specific Sampling Protocols

The following is a description of the sample collection procedures.

Collection Procedure for Fish Samples

Purpose

To collect fish samples which are representative (i.e., the same species and size ranges) of those harvested for subsistence purposes in the Villages of Seldovia, Nanwalek, Port Graham, and Tyonek Alaska. King (Chinook) salmon, red (sockeye) salmon, chum salmon, halibut, bullhead (sculpin), flounder, seabass and cod are the target species for collection, and will be analyzed for a suite of organic and inorganic contaminants.

Procedure

1. Identify the area (station) for collection of each composite sample. One composite sample of fish will be collected from each of three areas (stations) where village gatherers normally collect subsistence fish for red salmon, chum salmon, bullhead, halibut, seabass, flounder and cod. For king salmon, three composite samples will be collected from Tyonek, two composites will be collected from Port Graham, and one composite will be collected from Seldovia. The species to be collected at each village are as follows:

Seldovia: king salmon, red salmon, halibut and bullhead
Nanwalek: red salmon, halibut and cod
Port Graham: king salmon, chum salmon, seabass and flounder
Tyonek: king salmon and red salmon

The station where each composite sample is collected should be separate area if possible i.e., section of beach, area of a bay, etc. However, this may not be possible for all species collected within each village, where individual fish comprising a composite may be collected from different areas, or the same area at different times. Information should be carefully documented to indicate the collection locations and times for each composite sample.

2. Fish samples will be collected by set net and angling. Efforts should be made to minimize the use of tools or implements which may come in contact with the fish during collection (i.e., gaffs may be necessary to land some of the larger species). After the fish is landed or removed from a net, it will be killed (metal implements should not be used to kill fish, a clean wooden bat is recommended) and rinsed in ambient seawater at the collection site to remove sand particles and rock fragments.

3. For the king salmon and halibut, each fish will be placed in a heavy duty food grade polyethylene bag, and sealed with a cable tie. A completed sample ID label will be affixed to the cable tie and the bag will be placed inside another polyethylene bag and sealed with another cable tie. All other fish will be wrapped in aluminum foil (pre-rinsed with acetone and baked at 400 °C) before being "double bagged" and labeled as described above. For boat landed fish, extra care should be taken to keep the fish from contacting any sheen from the boat engine. At no time will the fish be allowed to contact

the bilge water in the boat. Clean nitrile gloves will be worn at all time during the cleaning and handling of the fish samples.

4. Each composite fish sample must be collected as 5 individual fish at each station. As noted previously, the "station" may encompass a large area (i.e., a bay), and the fish from one station may be collected on different days. For the Tyonek salmon, it is expected that the fish will be collected from set nets in one day for each species. The stations for Tyonek salmon collection should located such that each of the three composite samples are obtained from different areas of the set net beach (i.e., north area, middle area, and south area).

5. Field blanks will be collected during the fish sampling to determine the potential contribution of target contaminants from the sampling containers (polyethylene bags and foil) and shipping. A field blank will be prepared by filling a sampling bag with ~6 liters of commercially available distilled (DI) water and wrapping the bag in the same manner as the fish samples (i.e., double bags with sample ID label). The field blank will be frozen as soon as possible, and packaged and shipped in the same manner as the fish samples. Five fish field blanks will be collected during the course of the study: 1 with DI water only (Tyonek king salmon sample poly bags), 2 with DI water only (4 mil poly bags) and 2 with DI water and ~ 3 ft of rinsed and baked aluminum foil (4 mil poly bags).

Storage

During the collection of the composite fish sample, the individual fish will be stored on ice (a maximum of 24 hours). Within 24 hours the samples should be stored frozen (-20°C) until shipment. The fish samples from Tyonek will be stored frozen at 10th and M Seafood (Anchorage, Alaska) until shipment.

Documentation

1. For fish samples in polyethylene bags the sample ID label will be completed in permanent ink and affixed to the cable tie of the inner bag, no tape will be used to seal the bags.

2. A field record form should be completed for each composite fish sample. Information on the location of the station, sample identification, date and time of collection, collector's name(s), species, number of individuals, length of each individual, and a diagram of the collection site should be included on the field record form. Any additional information, as well as a log of daily sampling activities, should be noted in the field collection notebook.

3. Photo documentation will be collected from at least one sampling site for each species of fish collected. At a minimum, the photo documentation will include a picture of the collection site (station), a picture of a representative species, and a picture of the collection process.

Shipping

The fish samples will be shipped frozen to the designated laboratory(s) for chemical analysis. The fish will be packed in coolers for shipment. Excess space in the cooler will be filled with plastic "bubble wrap" to reduce the shifting of the samples during shipment. The cooler will be packed with dry ice (~20-30 lbs per cooler) to maintain a temperature below -4°C until receipt at the laboratory. A chain-of-custody form providing a listing of the sample ID of each sample included in the cooler will be filled out, signed and included in a sealed plastic bag in each cooler. After the final packing of each cooler for shipment (i.e. adding dry ice) the cooler will be sealed with fiberglass reinforced strapping tape, and a custody seal will be affixed across the lid of the cooler.

All coolers containing fish samples from Seldovia, Port Graham and Nanwalek will shipped via Federal Express Next Day Air from Homer Alaska. Federal Express will be notified of the shipment by 9:00 AM on the day of shipment, and will guarantee next-day delivery to Chicago, IL or San Diego, CA, if the samples are ready for pick-up in Homer by 12:00 noon. The fish samples from Tyonek will be shipped by 10th and M Seafood via Federal Express. The Tyonek fish will be shipped frozen in wax coated "fish boxes". The fish samples (which are "double bagged" in the field) will be wrapped in foil, packed in dry ice, and the boxes filled with "bubble wrap" for insulation. Chain-of-custody forms will be included in each fish box and a custody seal will be taped over the opening of each box prior to shipment.

Collection Procedure for Marine Plant Samples

Purpose

To collect the marine plant samples which are representative (i.e., the same species and size) of those harvested for subsistence purposes in the Villages of Nanwalek and Port Graham, Alaska. Goose tongue and kelp are the target species for collection, and will be analyzed for a suite of organic and inorganic contaminants.

Procedure

1. Identify the area (station) for collection of the composite sample. Three composite samples of each marine plant species will be collected from each of three areas (stations) where village gatherers normally collect the plants for subsistence. The station where each composite sample is to be collected should be separate area i.e., beach, bluff, outcrop, etc.

2. Plant samples should be picked by hand wearing clean nitrile gloves (no knives or tools should contact the plant during collection). After picking the plant the vegetation should be cleaned by hand (i.e., remove sand particles, grit, snails, etc.) wearing clean nitrile gloves in the same manner used to normally clean and prepare the plants for consumption.

3. After the vegetation is cleaned it should be rinsed in seawater at the collection site, and immediately placed in a pre-cleaned 1-L glass jar. The 1-L jars will be rinsed with ambient sea water prior to use. The process of picking and cleaning plants should be repeated until a minimum of ~300 g of vegetation is collected (for estimation, 300 g is approximately equivalent to a 1-L jar of tightly packed vegetation).

4. The composite plant sample must be collected from a minimum of 5 individual plants at each station. The number of individual plants used to comprise each composite sample should be noted on the field record form (if greater than 50 plants are required, an estimate of the number is sufficient).

5. Field blanks will be collected during the plant sampling to determine the potential contribution of target contaminants from the sampling containers (1-L glass jars) and shipping. A field blank will be prepared by rinsing a 1-L glass jar with ambient seawater, discarding the water and sealing the empty jar. The field blank will be frozen as soon as possible, and packaged and shipped in the same manner as the other plant samples. Five 1-L glass jar field blanks will be collected during the course of the study.

Storage

During the collection of the composite plant sample, the sample collection jar should be capped in stored in a cooler. After collection of composite sample is complete, the sample jar should be tightly sealed and stored on ice (a maximum of 24 hours). Within 24 hours the sample should be stored frozen (-20°C) until shipment.

Documentation

1. For each sample, a sample ID label will be completed in permanent ink and affixed to the sample jar. Clear cellophane tape will be placed over the label and completely around the jar.

2. A field record form should be completed for each individual composite plant sample. Information on the location of the station, sample identification, date and time of collection, collector's name(s), plant species, number of individual plants, and a diagram of the collection site should be included on the field record form. Any additional information, as well as a log of daily sampling activity, should be noted in the field collection notebook.

3. Photo documentation will be collected from at least one sampling site for each species of plant collected. At a minimum, the photo documentation will include a picture of the collection site (station), a picture of a representative plant, and a picture of the collection process.

Shipping

The plant samples will be shipped frozen to the designated laboratory(s) for chemical analysis. The plant samples will be packed in coolers for shipment. The glass jars will be placed in cardboard boxes and wrapped in plastic "bubble wrap" to reduce the possibility of breakage during shipment. The cooler will be packed with dry ice (~20 lbs per 60 quart cooler) to maintain a temperature below -4°C until receipt at the laboratory. A chain-of-custody form providing a listing of the sample ID of each sample included in the cooler will be filled out, signed and included in a sealed plastic bag in each cooler. After the final packing of each cooler for shipment (i.e. adding dry ice) the cooler will be sealed with fiberglass reinforced strapping tape, and a custody seal will be affixed across the lid of the cooler.

All coolers containing plant samples will shipped via Federal Express Next Day Air from Homer Alaska. Federal Express will be notified of the shipment by 9:00 AM on the day of shipment, and will guarantee next-day delivery to Chicago, IL or San Diego, CA, if the samples are ready for pick-up in Homer by 12:00 noon.

Collection Procedure for Invertebrate Samples

Purpose

To collect invertebrate samples which are representative (i.e., the same species and size ranges) of those harvested for subsistence purposes in the Villages of Seldovia, Nanwalek, Ninilchik, and Port Graham, Alaska. Butter clams, mussels, chitons, snails, octopus and razor clams are the target species for collection, and will be analyzed for a suite of organic and inorganic contaminants.

Procedure

1. Identify the area (station) for collection of each composite sample. Three composite samples of invertebrate species (with the exception of octopus samples which will be comprised of one individual) will be collected from each of three areas (stations) where village gatherers normally collect the invertebrates for subsistence. The species to be collected at each village are as follows:

Seldovia: butter clams, mussels, chitons, snails
Nanwalek: butter clams, chitons, octopus, snails
Ninilchik: razor clams
Port Graham: butter clams, chitons, octopus, snails

The station where each composite sample is to be collected should be separate area i.e., beach , bluff, outcrop, etc.

2. Invertebrate samples will be collected by hand wearing clean nitrile gloves. Efforts should be made to minimize the use of tools or implements during collection (i.e., clam rakes may be necessary for clam collection, jigs may be used for octopus collection). After picking or digging the invertebrates, the individual organisms should be rinsed in ambient seawater at the collection site to remove sand particles and rock fragments. Clean nitrile gloves will be worn at all time during the cleaning and handling or the organisms.

3. After the individual organisms have been cleaned they will be immediately placed in a pre-cleaned 1-L glass jar. The process of picking/digging and cleaning invertebrates will be repeated until a minimum of ~300 g of is collected (for estimation, 300 g is approximately equivalent to a 1-L jar of tightly packed clams, chitons, mussels and snails).

4. The composite invertebrate sample must be collected from a minimum of 5 individual organisms at each station. The number of individual used to comprise each composite sample should be noted on the field record form (if greater than 50 invertebrates are required, an estimate of the number is sufficient).

5. The one exception is octopus, which will not be comprised as a composite sample, due to expected availability. For octopus, one individual will represent the

sample for a given station. Octopus samples will be handled in nitrile gloves and rinsed in ambient seawater before being placed in a 1-L jar. If an individual octopus will not fit into a sample jar, the octopus will be placed in a heavy duty food grade polyethylene bag, and sealed with a cable tie. A sample completed sample ID label will be affixed to the cable tie and the bag will be placed inside another polyethylene bag and sealed with another cable tie.

5. Field blanks will be collected during the invertebrate sampling to determine the potential contribution of target contaminants from the sampling containers (1-L glass jars) and shipping. 5. A field blank will be prepared by rinsing a 1-L glass jar with ambient seawater, discarding the water and sealing the empty jar. The field blank will be frozen as soon as possible, and packaged and shipped in the same manner as the other invertebrate samples. Five 1-L glass jar field blanks will be collected during the course of the study

Storage

During the collection of the composite invertebrate sample, the sample collection jar should be capped in stored in a cooler. After collection of the composite sample is complete, the sample jar should be tightly sealed and stored on ice (a maximum of 24 hours). Within 24 hours the sample should be stored frozen (-20°C) until shipment.

Documentation

1. For each sample, a sample ID label will be completed in permanent ink and affixed to the sample jar. Clear cellophane tape will be placed over the label and completely around the jar. For samples in polyethylene bags (i.e., octopus) the sample ID label will be affixed to the cable tie, and no tape will be used.

2. A field record form should be completed for each individual composite invertebrate sample. Information on the location of the station, sample identification, date and time of collection, collector's name(s), invertebrate species, number of individuals, and a diagram of the collection site should be included on the field record form. Any additional information, as well as a log of daily sampling activities, should be noted in the field collection notebook.

3. Photo documentation will be collected from at least one sampling site for each species of invertebrate collected. At a minimum, the photo documentation will include a picture of the collection site (station). a picture of a representative species, and a picture of the collection process.

Shipping

The invertebrate samples will be shipped frozen to the designated laboratory(s) for chemical analysis. The invertebrate samples will be packed in coolers for shipment. The glass jars will be placed in cardboard boxes and wrapped in plastic "bubble wrap" to reduce the possibility of breakage during shipment. The cooler will be packed with dry ice (~20 lbs per 60 quart cooler) to maintain a temperature below -4°C until receipt at the laboratory. A chain-of-custody form providing a listing of the sample ID of each sample included in the cooler will be filled out, signed and included in a sealed plastic bag in each cooler. After the final packing of each cooler for shipment (i.e. adding dry ice) the cooler will be sealed with fiberglass reinforced strapping tape, and a custody seal will be affixed across the lid of the cooler.

All coolers containing invertebrate samples will shipped via Federal Express Next Day Air from Homer Alaska. Federal Express will be notified of the shipment by 9:00 AM on the day of shipment, and will guarantee next-day delivery to Chicago, IL or San Diego, CA, if the samples are ready for pick-up in Homer by 12:00 noon.

Collection Procedure for Mammal Samples

Purpose

To collect mammal samples which are representative of those harvested for subsistence purposes in the Villages of Nanwalek and Tyonek Alaska. Mammal samples will be collected as part of the local village seal and beluga whale hunts and will be analyzed for a suite of organic and inorganic contaminants.

Procedure

1. Beluga whale samples will be collected from Tyonek and seal samples will be collected from Tyonek and Nanwalek. The whale sample will be collected by NMFS following the NMFS sampling procedure. The seal samples will be collected by ADF&G in conjunction with the HSC following the ADF&G sampling procedure. Information should be carefully documented to indicate the collection locations and times for each whale and seal sample.

2. For both whale and seal three tissues will be sampled from each individual. The tissue types are liver, blubber, and meat. Samples of each tissue type (~500 g of tissue) will be wrapped in aluminum foil (pre-rinsed and baked - seal samples only) and placed in a heavy duty food grade polyethylene bag (seal samples) or teflon bag (beluga samples), and sealed with a cable tie. A completed sample ID label will be affixed to the cable tie and the bag will be placed inside another polyethylene bag and sealed with another cable tie.

3. Efforts will be made to reduce the contact of metal implements during the sub-sampling of the mammal tissues. If possible only teflon knives will used to collect the samples; however, it is expected that clean stainless steel knives may be used during the collection of the tissues. Tissue samples will not be collected from the immediate area where the mammal was shot or speared to reduce the potential of metals contamination. Clean nitrile gloves will be worn at all times during the collection and handling of the whale and seal tissue samples.

4. Field blanks will be collected during the mammal sampling to determine the potential contribution of target contaminants from the sampling containers (polyethylene or teflon bags) and shipping. A field blank will be prepared by filling a sampling bag with ~6 liters of commercially available distilled water and wrapping the bag in the same manner as the mammal samples (i.e., double bags with sample ID label). The field blank will be frozen as soon as possible, and packaged and shipped in the same manner as the mammal samples. Two mammal field blanks will be collected during the course of the study (one for teflon bags and one for polyethylene bags).

Storage

During the collection of the whale and seal samples, the individual tissue samples will be stored on ice (a maximum of 24 hours). Within 24 hours the samples should be stored frozen (-20°C) until shipment.

Documentation

1. For mammal samples the sample ID label will be completed in permanent ink and affixed to the cable tie of the inner bag, no tape will be used to seal the bags.

2. Information on the location of the station, sample identification, date and time of collection, collector's name(s), species, tissue type, and a diagram of the collection site should be included in the field collection notebook.

3. Photo documentation will be collected from at least one sampling site for each mammal species collected. At a minimum, the photo documentation will include a picture of the collection site (station), a picture of a representative species, and a picture of the collection process.

Shipping

The mammal samples will be shipped frozen to the designated laboratory(s) for chemical analysis. The mammal tissue samples will be packed in coolers for shipment. Excess space in the cooler will be filled with plastic "bubble wrap" to reduce the shifting of the samples during shipment. The cooler will be packed with dry ice (~20 lbs per cooler) to maintain a temperature below -4°C until receipt at the laboratory. A chain-of-custody form providing a listing of the sample ID of each sample included in the cooler will be filled out, signed and included in a sealed plastic bag in each cooler. After the final packing of each cooler for shipment (i.e. adding dry ice) the cooler will be sealed with fiberglass reinforced strapping tape, and a custody seal will be affixed across the lid of the cooler.

All coolers containing mammal samples from will shipped via Federal Express Next Day Air from Anchorage Alaska. Federal Express will be notified of the shipment by 9:00 AM on the day of shipment, and will guarantee next-day delivery to Chicago, IL or San Diego, CA, if the samples are ready for pick-up by 12:00 noon.

2.3 Sample Handling and Custody Requirements (B3)

Sample handling procedures are described in the individual sampling protocols above. Other general provisions for sample handling and custody are described herein.

As soon as possible after collection (or after transfer of sample from collector), the field sampling team will begin the process identifying, labeling, packaging, and storing the sample(s). Identification of the sample composites and individual samples that make up the composite is made through the use of sample labels and the field records.

2.3.1 Naming Scheme

Samples will be identified and tracked with a unique naming scheme. Samples will be assigned an identification number by the field sampling staff using the following format:

VC-SP-nn-mm

where,

VC = Village Code
SP = Species Type
nn = Station Number, a unique sequential number assigned in field
mm = Individual Number, a unique sequential number assigned in field (if individuals are labeled; if not omit this field)

For example "SE-RS-01-02" would be assigned to a Seldovia red salmon collected at station 01, individual fish 02 from that composite.

2.3.2 Sample Labeling

Each sample will be physically identified by the use of a sample label (Figure 5). Instructions for completion of sample labels are described below. For plant and invertebrate samples, which are composited during collection, the completed sample label is affixed to the sample jar. For fish samples, each individual fish sample that comprises the composite is labeled by affixing a completed sample label to the outside of the inner sample packaging. All entries on the sample labels will be made with indelible ink.

2.3.3 Field Records

Detailed documentation of the samples is recorded on Field Record forms (Figures 2, 3, and 4). These forms record specific details about the samples and the collection location. One form must be completed for each sample composite. The record for fish samples includes documentation of the collection of the individual samples that comprise the composite.

Each form will include a diagram of the location of the sample collection points. Relevant notes from field notebooks made during collection should be transcribed to the Field Record.

Upon completion, a copy of the Field Record is made to accompany samples during shipment. The original should be retained by the field sample team for transfer to the ADL Project Manager for final disposition.

2.3.4 Chain-of-Custody

All individual samples and sample composites will be transferred to the receiving laboratory under chain-of-custody. The chain-of-custody form acts as a record of sample shipment and a catalog of the contents of each shipment.

The chain-of-custody form is shown in Figure 6.

2.4 Analytical Methods Requirements (B4)

Samples will be shipped to locations designated by the USEPA Project Manager for analytical testing. Analytical testing is outside the scope of this QAPP and is therefore not further addressed.

2.5 Quality Control Requirements (B5)

Laboratory quality control requirements are outside the scope of this document. However, to monitor the potential contamination of samples from the sample containers, shipping containers, packing and the shipping process field blanks will be prepared in the field and shipped with the samples to the analytical laboratory.

The field blanks for invertebrates and plant samples will consist of a pre-cleaned 1-L glass jar which is rinsed with ambient seawater and frozen in the same manner as the samples. Five field blanks for plant and invertebrate samples will be collected during the study.

The field blanks for fish and mammals will be prepared by filling a polyethylene sampling bag (teflon bag for beluga samples) with ~6 liters of commercially available distilled water and wrapping the bag in the same manner as the fish and mammal samples (i.e., double bags with sample ID label). The field blanks will be frozen as soon as possible, and packaged and shipped in the same manner as the fish and mammal samples. Five fish field blanks will be collected during the course of the study: 1 with DI water only (Tyonek king salmon sample poly bags), 2 with DI water only (4 mil poly bags) and 2 with DI water and ~ 3 ft of rinsed and baked aluminum foil (4 mil poly bags). Two mammal field blanks will be collected during the course of the study (one for teflon bags and one for polyethylene bags).

In addition to the field blanks, two samples of the DI water used to prepare the field blanks (~6 L each) will be frozen in the original container and sent to the analytical laboratory.

2.6 Instrument and Equipment Testing, Inspection, and Maintenance Requirements (B6)

Not applicable.

2.7 Instrument Calibration and Frequency (B7)

Not applicable.

2.8 Inspection/Acceptance Requirements for Supplies and Consumables (B8)

Because of the remote location of the sampling area, careful and thorough planning is required to efficiently and effectively conduct the sample collection tasks. A basic checklist of field sampling equipment and supplies is given in Table 1. This checklist should be reviewed prior to each sampling event.

Table 1: Field Sampling Equipment and Supplies Checklist

Boat supplies
Fuel supply (primary and auxiliary supply)
Spare parts repair kit
Life preservers
First aid kit with emergency phone numbers
Spare oars
Nautical charts of sampling site locations
Sample collection equipment
Record keeping and documentation supplies
Field notebooks
Sample identification labels
Field Record forms
Chain-of-custody forms
Indelible ink pens
Sample processing supplies
Aluminum foil
Distilled water
Cable ties
Several sizes of food grade polyethylene bags
Resealable plastic bags for storage of collection documentation
Sample preservation and shipping supplies
Ice
Dry ice
Coolers
Filament reinforced tape to seal ice chests
Ice chest chain-of-custody seals

The selection of the equipment must be made with consideration of its potential to contaminate samples during collection or handling. As such, laboratory grade supplies should be preferred over lessor quality materials. Food grade materials can in some cases provide an acceptable alternative.

2.9 Data Acquisition Requirements (Non-direct Measurements) (B9)

Measurements of sampling locations (longitude and latitude) will be made from current NOAA charts of the sampling area.

2.10 Data Management (B10)

Samples are tracked using the field records, field notebooks, and chain-of-custody forms. Because the sampling effort is a cooperative one involving a number of local sampling teams, the diligence of the field staff in completion of the proper records is essential.

Upon completion of the sampling effort, a database of samples and sampling locations will be constructed by the Arthur D. Little Project Manager from the project documentation. This data summary will be included in the final project report.

To track shipments of sample coolers, only shipping services which include tracking of packages (e.g., FedEx) will be used.

3. Assessment and Oversight (Group C)

This group of QAPP elements addresses the activities for assessing the effectiveness of the implementation of the project and associated QA/QC.

3.1 Assessments and Response Actions (C1)

To allow for oversight, the Arthur D. Little Project Manager will communicate the status of the project with the USEPA Project Manager at a minimum every other day.

There will also be a daily communication between the Arthur D. Little Project Manager and the field sampling teams when sampling is active.

To provide an initial assessment, the USEPA Project Manager will supervise the sampling activities during the first week of activity.

3.2 Corrective Actions

Issues requiring corrective action or changes to the project requirements or scope initiated by the field sampling teams are directed to the Field Sampling Task Leader then to the Arthur D. Little Project Manager. The Arthur D. Little Project Manager will then communicate with the USEPA Project Manager for design and approval of the appropriate action.

3.3 Reports to Management (C2)

Upon completion of the field activities, the project will be summarized in report form by the Arthur D. Little Project Manager. The report will be issued in draft form to the USEPA Project Manager for review and comment. Comments from USEPA will be incorporated into the report which will then be issued in final form.

4. Data Validation And Usability (Group D)

This group of QAPP elements covers the QA activities that occur after the data collection phase of the project is completed.

4.1 Data Review, Validation, and Verification Requirements (D1)

All field notes and records will be reviewed by the Arthur D. Little Project Manager and QA Manager for completeness and correctness.

4.2 Validation and Verification Methods (D2) and Reconciliation With User Requirements (D3)

The Arthur D. Little Project Manager and QA Manager will review all COC and field records from the sampling effort. Any discrepancies in field records will be reconciled with the associated field sampling personnel and communicated to the USEPA Project Manager.

Figure 2: Field Record for Fish Species

Field Record for Cook Inlet Contaminant Study Sampling - Fish Species

Composite Sample ID: ____________________

Sampling Date and Time: ____________________

Site Location

Site Name: ____________________

Site Description: ____________________

Collection Method: ____________________

Collector Name (print and sign): ____________________

Affiliation: ____________________ Phone: ____________________

Address: ____________________

Sample Description

Fish Species Name: ____________________ Number of Individuals: ____________________

Fish #	Length (mm)	Location	Date/Time	Notes
01				
02				
03				
04				
05				

Additional Notes: ____________________

minimum individual size should be no less than 75% maximum individual size

Sampling Site Diagram

Figure 3: Field Record for Plant Species

Field Record for Cook Inlet Contaminant Study Sampling - Plant Species

Composite Sample ID: ______________________________

Sampling Date and Time: ______________________________

Site Location

Site Name: ______________________________

Site Description: ______________________________

Collection Method: ______________________________

Collector Name (print and sign): ______________________________

Affiliation: ______________________ Phone: ______________

Address: ______________________________

Sample Description

Plant Species Name: ______________________ Number of Individuals: ________

Additional Notes: ______________________________

____

____

____

____

individual plants for composite should be of similar size and of a size normally collected for subsistence

Sampling Site Diagram

Figure 4: Field Record for Invertebrate Species

Field Record for Cook Inlet Contaminant Study Sampling - Invertebrate Species

Composite Sample ID: ______________________________

Sampling Date and Time: ______________________________

Site Location

Site Name: ______________________________

Site Description: ______________________________

Collection Method: ______________________________

Collector Name (print and sign): ______________________________

Affiliation: ______________________ Phone: ______________

Address: ______________________________

Sample Description

Plant Species Name: ______________________ Number of Individuals: __________

Additional Notes: ______________________________

individual invertebrates for composite should be of similar size and of a size normally collected for subsistence

Sampling Site Diagram

Figure 5: Example Sample Label

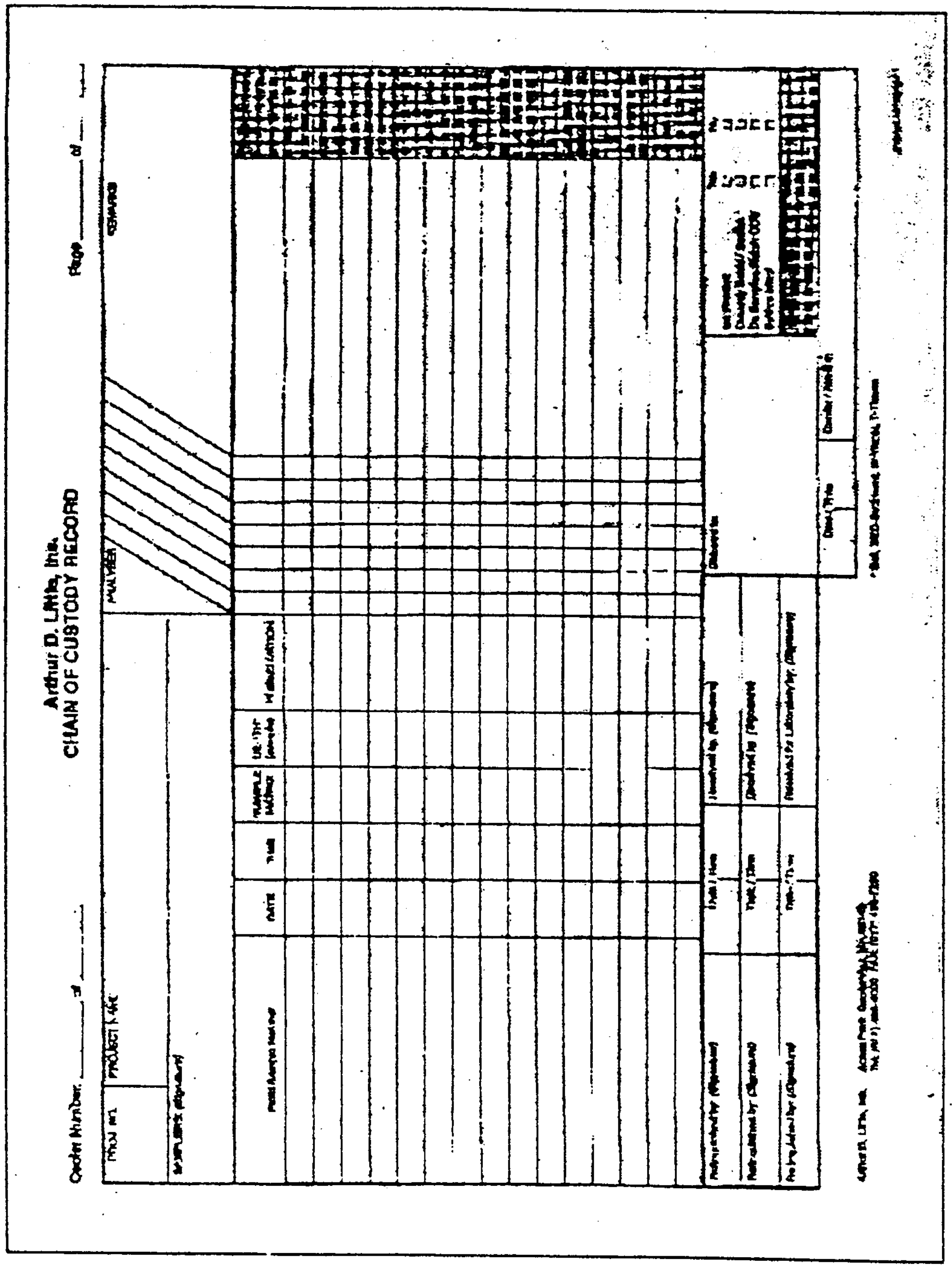
I-CHEM

CLIENT/SOURCE	GRAB COMPOSITE
SITE NAME	DATE/TIME
SAMPLE #	PRESERVATIVE
ANALYSIS	COLL BY

KEY:

CLIENT/SOURCE - enter "COOK INLET STUDY"
GRAB or COMPOSITE - check GRAB if the sample is an individual; check COMPOSITE if the sample is a composite
SITE NAME - identify the Village related to the harvest subsistence area
DATE/TIME - note the date and time from the Field Record
SAMPLE # - record the composite sample ID from the Field Record; include individual number if the sample is an individual
PRESERVATIVE - enter "frozen"
ANALYSIS - enter N/A
COLL BY - record the collector initials

Figure 6: Chain-of-Custody Record



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Quality Assurance Project Plan
for
Sample Homogenization, Compositing, and Analysis
in the
Cook Inlet Contaminant Study

October 1997

Prepared by

United States Environmental Protection Agency
Office of Water
Office of Science and Technology

TABLE OF CONTENTS

1.0 Introduction and Scope

This Quality Assurance Project Plan (QAPP) presents the measurement quality objectives (MQOs) established for the analysis of environmental samples collected during EPA's 1997 Cook Inlet Contaminant Study. This QAPP also describes the methods and procedures that will be followed to ensure that these MQOs are met. This document addresses only the sample analysis effort; data quality objectives and procedures related to sample collection are described in a separate QAPP.(1)

This QAPP was prepared in accordance with and contains each of the elements described in *EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations*, EPA QA/R-5, Draft Interim Final, August 1994. To improve clarity, the order of certain elements in this QAPP has been modified slightly from the order presented in EPA QA/R-5. For example, the Background section (element "A5" in EPA QA/R-5) is presented before the Project Organization section (element "A4" in RPS QA/R-5) in this QAPP.

In accordance with the guidance provided in EPA QA/R-5, this QAPP is considered to be a dynamic document that is subject to change as sample collection and analysis progresses during the Cook Inlet Contaminant Study. All changes to procedures described in this QAPP will be reviewed by the EPA Analytical Project Manager and the EPA Quality Assurance Manager to determine if the changes signficantly impact the technical and quality objectives of the project. If changes are deemed to be significant, the QAPP will be revised accordingly.

2.0 Project Background

The Cook Inlet Contaminant Study is being conducted by the EPA Office of Science and Technology (OST) as part of its effort to assess health risks to Native American tribes living in coastal Alaska. This human health risk assessment is part of a larger OST effort to characterize human health risks from pollutants associated with Offshore Oil and Gas Industry practices.

3.0 Project Organization

The Office of Science and Technology is responsible for overall management of the study; day-to-day responsibility for managing various aspects of the study have been delegated to the Standards and Applied Science Division (SASD) and the Engineering and Analysis Division (EAD) within OST. SASD is responsible for managing all sample collection and data analysis activities associated with this study; EAD is responsible for managing all laboratory analysis and data verification (data review) activities. Both EAD and SASD are responsible for day-to-day interaction with contractors and with other federal, state, and local authorities involved in the project.

Several federal, state, local, and contractor organizations are participating in this project. These include EPA, the National Marine Fisheries Service (NMFS), the Alaska Department of Fish and Game (AF&G), the Harbor Seal Commission (HSC), DynCorp Information & Engineering Technology (DynCorp), Arthur D. Little Inc. (ADL), and several Village Coordinators from the villages of Tyonek, Seldovia, Port Graham, and Nanwalek, Alaska. Figure 1 illustrates the relationships and lines of communications between each of these organizations.

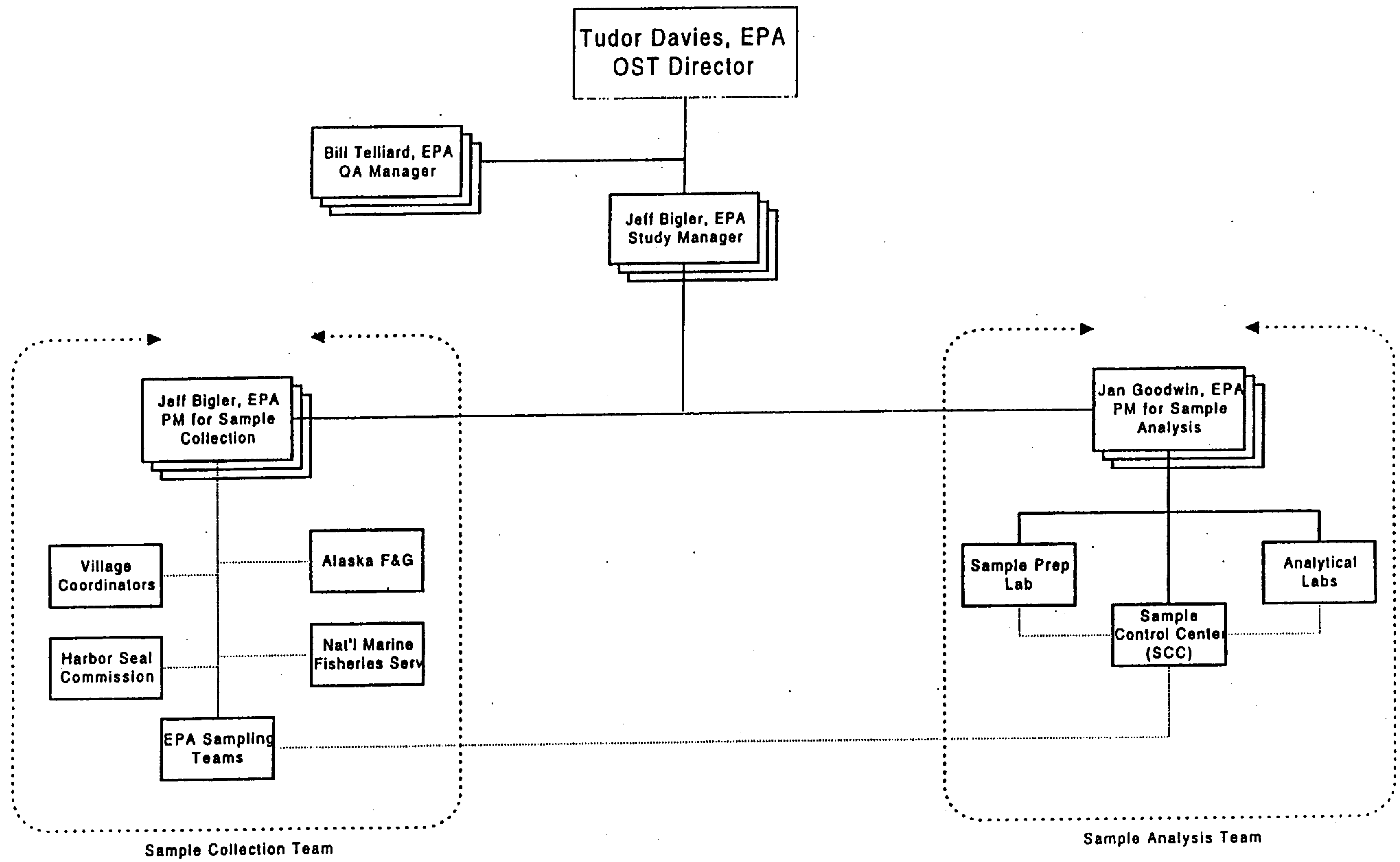


Figure 1 - Project Organization

Sections 3.1 through 3.3 describe the roles and responsibilities of the individuals involved in the shipment, tracking, homogenization, compositing, and analysis of samples under this study. (Roles and responsibilities of those organizations involved solely in the sample planning and collection effort are described in a separate document.)[1] Specifically, Section 3.1 describes the responsibilities of EPA staff; Section 3.2 describes the responsibilities of contractor staff at the Sample Control Center (operated by DynCorp), and Section 3.3 describes the responsibilities of staff at each of the contract laboratories that will support this study.

3.1 EPA Staff

3.1.1 OST Director

The OST Director, Tudor Davies, is responsible for providing financial and staff resources necessary to meet study objectives and implement study requirements described in this QAPP.

3.1.2 OST QA Manager

The OST QA Manager, William Telliard, is responsible for assisting the EPA Study Manager and Project Managers with the development and implementation of QAPPs for this study. The QA Manager also is responsible for ensuring that all QA procedures described in this QAPP are followed, reporting any deviations from this QAPP to the Project Managers, and assisting the Project Managers in implementing corrective actions necessary to resolve these deviations. The QA Manager reports directly to the OST Director.

3.1.3 OST Study Manager

The OST Study Manager, Jeff Bigler, reports directly to the OST Director and is responsible for providing overall direction concerning the study to the EPA Project Managers shown in Figure 1. The OST Study Manager also is responsible for:

- Developing and implementing an overall QAPP applicable to all phases of the Cook Inlet Contaminant Study
- Communicating study objectives to the EPA Project Managers shown in Figure 1
- Reviewing and approving all major work products associated with the study
- Participating in meetings with the EPA Project Managers, other EPA staff, and staff from other organizations and contractors concerning the study
- Working with the OST QA Manager to identify corrective actions necessary to ensure that study objectives are met.

3.1.4 OST Project Manager for Sample Analysis

The OST Project Manager for Sample Analysis, Jan Goodwin, reports directly to the OST Director, and is responsible for:

- Developing and implementing this QAPP

- Daily oversight of EPA and contractor staff involved in activities related to analysis of samples collected in this study
- Communicating project objectives to all EPA and contractor staff involved in the analysis of samples collected in this study
- Reviewing and approving major deliverables related to the analysis of samples collected in this study
- Participating in meetings with the OST Project Manager for Sample Collection, the OST QA Manager, and the OST Director concerning study objectives, schedules, and concerns
- Providing technical assistance concerning sample analysis and data evaluation to the OST Project Manager for Sample Collection

3.2 *DynCorp Sample Control Center Staff*

3.2.1 SCC Program Manager

The SCC Program Manager, Jim King, is responsible for aquiring and applying corporate resources needed to ensure that project deliverables are completed on time, within budget and to client satisfaction.

3.2.2 SCC Project Manager

The SCC Project Manager, Lynn Riddick, will be responsible for developing and obtaining approval of this QAPP and for ensuring that the practices required in this QAPP meet or exceed those outlined in Quality Management Plan approved for use in DynCorp's Sample Control Center (SCC) contract. The Project Manager reports directly to the SCC Program Manager (Jim King) and the EPA Analytical Project Manager (Jan Goodwin). Other responsibilities of the SCC Project Manager include:

- Working with the EPA Analytical Project Manager to define project objectives and develop a project schedule
- Acquiring and applying corporate resources needed to ensure that project deliverables are completed on time, within budget, and to client satisfaction
- Communicating project objectives to the SCC Study Coordinator
- Monitoring performance of the SCC Study Coordinator and other SCC staff participating in the Cook Inlet Contaminant Study to ensure the quality, timeliness, and responsiveness of work performed
- Reviewing and approving major study deliverables
- Participating in meetings with EPA staff concerning study objectives, schedules, and concerns.

3.2.3 SCC QA Manager

The SCC QA Manager, Maggie Jones, is independent the SCC project and reports directly to DynCorp Vice President, Don Trees. Ms. Jones is responsible for:

- Assisting the SCC Project Manager with the development and implementation of this QAPP
- Ensuring that all QA procedures described in this QAPP are followed during the Cook

Inlet Contaminant Study
- Reporting deviations from this QAPP to the Project Manager and assisting the Project Manager in implementing corrective actions to resolve these deviations.

3.2.4 SCC Study Coordinator

The SCC Study Coordinator (Carrie Buswell) reports directly to the SCC Project Manager The SCC Study Coordinator is responsible for:

- Developing and implementing any SOPs necessary to meet the objectives of the Cook Inlet Contaminant Study
- Day-to-day oversight of technical activities performed by SCC staff participating in the Cook Inlet Contaminant Study
- Communicating study objectives and requirements to the SCC Scheduling Coordinators, the SCC Data Review Team Leader, and the Database Administrator
- Supervising daily activities of the SCC Scheduling Coordinators, Data Reviewers, and Database Administrator and assisting these individuals with the resolution of problems that arise during study implementation
- Notifying the Project Manager of anticipated difficulties in meeting project schedules or other requirements
- Identifying problems at the task level and resolving difficulties in consultation with the SCC Project Manager and the EPA Analytical Project Manager
- Ensuring that all necessary corrective action procedures are documented and implemented in a timely manner
- Adhering to the schedule approved for this study
- Reviewing all deliverables prior to submission

3.2.5 SCC Scheduling Coordinators

A primary SCC Scheduling Coordinator (Janet Trent-Wilkinson) will be assigned to the Cook Inlet Contaminant Study. This coordinator will report directly to the SCC Study Coordinator and will be supported by a back-up Scheduling Coordinator. Responsibilities of the SCC Scheduling Coordinators include:

- Understanding and implementing all sample scheduling and tracking requirements described in this QAPP
- Documenting and notifying the Study Coordinator of problems related to the scheduling, shipment, and tracking of samples and data
- Working with the Study Coordinator to identify and implement corrective actions for such problems
- Day-to-day communication and coordination with field sampling teams and laboratory personnel during sample shipment and laboratory analysis
- Preparing and submitting to the Study Coordinator reports that summarize the status of sample analysis, data reporting, and data evaluation activities
- Entering information in and updating the automated sample and data tracking system in a timely manner

3.2.6 SCC Data Reviewer Manager

The SCC Data Review Manager (MaryAnne Templeton) reports directly to the SCC Study Coordinator and is responsible for:

- Understanding and communicating to SCC Data Reviewers all Cook Inlet Contaminant Study requirements concerning data quality objectives, measurement quality objectives, data quality audits, data quality assessments, and data management.
- Supervising day-to-day activities of SCC Data Reviewers supporting the Cook Inlet Contaminant Study and working with the Data Reviewers and the Study Coordinator to resolve problems and implement corrective actions in a timely manner
- Ensuring that SCC Data Reviewers adhere to all data quality audit and assessment requirement requirements, including documentation, corrective action, and database development procedures, described in this QAPP
- Immediately notifying the Study Coordinator of technical or resource limitations that adversely affect study schedules or requirements
- Performing a technical review of all SCC deliverables pertaining to data quality audits and assessments before submission to the SCC Study Coordinator for final review

3.2.7 SCC Data Reviewers

Several SCC chemists will be assigned to perform data quality audits and data quality assessments as described in this QAPP. These SCC Data Reviewers will report directly to the SCC Data Review Manager and are responsible for:

- Understanding and implementing the data quality audit and assessment requirements described in this QAPP.
- Immediately notifying the SCC Data Review Manager of technical difficulties or other problems that adversely impact study schedules
- Working with the analytical laboratories to implement corrective actions necessary to resolve data quality problems in a timely manner and to maximize the amount of usable data generated in this study
- Documenting all problems and corrective actions related to data quality
- Preparing written narratives that describe the overall quality of each laboratory data submission and recommendations concerning data usability
- Updating the automated sample and data tracking system in a timely manner

3.2.8 SCC Database Administrator

The SCC Database Administrator (John DeHart) will report directly to the SCC Study Coordinator and is responsible for:

- Understanding and implementing all database development and maintenance requirements described in this QAPP
- Working with SCC Data Reviewers, the SCC Data Review Manager, and the SCC Study Coordinator to identify problems and implement corrective actions in a timely manner
- Notifying the SCC Study Coordinator of technical or resource constraints that may adversely impact study schedules

- Documenting database problems and corrective actions in a manner that is both timely and consistent with this QAPP
- Preparing written database status reports as requested by the SCC Study Coordinator.

3.3 Contract Laboratories

All chemical analyses performed under this study will be performed by laboratories operating under existing long-term contracts known as the "megalab" contracts. These contracts provide EPA with a mechanism to issue short-term delivery orders for a wide range of analytical services on an as-needed basis. Each contract stipulates that the laboratory, and any subcontract laboratory utilized by the prime contract laboratory, maintain and adhere to an EPA-approved QAPP. Each contract further stipulates that a Project Manager, a Quality Assurance Manager, and certain laboratory staff be available and dedicated to each project. Sections 3.3.1 through 3.3.6 below describe the responsibilities of each of these staff members.

3.3.1 Laboratory Project Manager

The laboratory Project manager is responsible for the overall technical activities under the megalab contract. This individual is responsible for planning, conducting, and supervising projects of major significance (such as the Cook Inlet Contaminant Study), and for supplying technical advice and counsel to other professionals.

3.3.2 Laboratory QA Manager

This individual is responsible for quality assurance (QA) of all technical efforts performed under the contract, and reports directly to senior management. This individual directs assistance, reviews progress and evaluates results, and makes changes in methods, design or equipment where necessary.

3.3.3 Sample Custodian

The Sample Custodian is responsible for logging in, handling, and storing samples.

3.3.4 Sample Preparation Laboratory Supervisor, GC/MS Laboratory Supervisor, HPLC Laboratory Supervisor, and Inorganic Laboratory Supervisor

These individuals responsible for all technical efforts performed in support of the megalab contract within the Sample Preparation, GC/MS, HPLC, and Inorganic Laboratories. They are responsible for supervising the laboratory staff who prepare and analyze samples under the megalab contract.

3.3.4 GC/MS Operators, Mass Spectral Interpretation Specialists, Purge and Trap Specialists, Extraction Specialists, Inductively Coupled Plasma Spectroscopists, Atomic Absorption Spectroscopists, and Sample Prep Specialists

These individuals are responsible for sample preparation, sample extraction, and sample analysis on a daily basis. They also are responsible for adhering to the analytical and QA/QC requirements specified in the megalab contract and for reporting to their supervisors any difficulties encountered.

3.3.5 Systems Manager and Programmer Analysts

These individuals are responsible for developing and maintaining any automated systems necessary to report analytical results produced under the megalab contract and for ensuring that these systems report data in a manner that is consistent with contract requirements.

3.3.6 Data Reporting and Delivery Officer

This individual is responsible for the organization, packaging, copying, and delivery of data deliverables submitted under the megalab contract.

4.0 Project Description

Under this project, marine plant, marine mammal, marine invertebrate, and fish samples will be collected from areas near four villages around Cook Inlet, Alaska: Tyonek, Seldovia, Port Graham, and Nanwalek. Activities pertaining to the collection of samples (e.g., decisions and procedures related to sampling design, sample locations, sample types, and sample collection techniques) are described in the "Combined Workplan and Quality Assurance Project Plan for the Cook Inlet Contaminant Study Sampling" (June 5, 1997) and are not discussed here. This QAPP describes decisions and procedures related to the shipment, storage, and laboratory analysis of the samples collected in this study.

4.1 Contractor Support

EAD will obtain laboratory services for the preparation and analysis of samples through its "megalab" contracts . It is anticipated that at least two contract laboratories will be involved in the study. One contract laboratory will be responsible for all activities related to the homogenization, compositing, and/or aliquotting of samples. It is possible that this laboratory also will be issued a delivery order to perform some or all of the subsequent sample analyses. If necessary to maximize expertise and minimize costs, EPA also may issue delivery orders to one or more additional contract laboratories.

The Sample Control Center (SCC, operated by DynCorp under EPA Contract 68-C3-0337) will be responsible for facilitating effective communication among all of the parties involved in the shipment and analysis of samples under this study. SCC also will be responsible documenting all sample shipments, problems that arise, and resolutions to those problems. Finally, SCC will be responsible for reviewing laboratory data to ensure that the measurement quality objectives (MQOs) detailed in Section 5 of this QAPP are met, working with the laboratories and EPA to correct QC failures, where possible, and for documenting the extent to which data submissions meet study MQOs.

4.2 Project Summary

Samples will be collected during Summer, 1997, as described in the "Combined Workplan and Quality Assurance Project Plan for the Cook Inlet Contaminant Study Sampling" (June 5, 1997). Following collection, the samples will be frozen and shipped to a contract laboratory (the "Sample Prep Lab" for homogenization, compositing, and/or aliquotting. This laboratory must have the capability to store, homogenize, composite, and otherwise handle biological and aqueous samples for low level organic and inorganic analysis without contaminating the samples. To verify that these procedures are contaminant-free, the Sample Prep Lab will prepare and analyze equipment blanks with each batch of field samples prepared. These blanks will be analyzed for each of the target analytes listed in Table 1 of this QAPP. If the Sample Prep Lab does not have the analytical capability to analyze for all of these target pollutants, it will ship the equipment blanks to a qualified laboratory, operating under an approved EPA contract or subcontract, for analysis of the required pollutants. Data demonstrating the absence of contamination will be sent to SCC for verification.

Upon completion of these activities, the Sample Prep Lab will ship frozen aliquots of the individual or composited samples to other contract or subcontract laboratories for analysis. Instructions regarding shipment of these samples (e.g., dates, recipients, etc) will be provided by SCC. EPA anticipates that at least two analytical laboratories will be needed to obtain results for all of the target analytes listed in Table 1. Analytical laboratories will use the methods described in this QAPP and specified in delivery orders issued under existing EPA contracts to prepare and analyze the samples.

Following sample shipment, SCC will maintain routine communication with the analytical laboratories to identify and communicate to EPA any significant questions, problems, or delays that arise during the course of sample analysis. Upon completion of sample analysis, the laboratories will forward data packages containing the results of all field and QC sample analyses to SCC for review. SCC will review the data package to ensure that the procedures followed and the results obtained are consistent with those specified by EPA. To the extent possible, SCC will work with the laboratory to correct any identified problems prior to submitting the final, reviewed data packages to EPA. SCC also will evaluate overall data quality against the MQOs established in this QAPP and will prepare a written assessment of its findings. SCC will include this written assessment with the final, reviewed data packages forwarded to EPA. Upon receipt of the reviewed data packages and SCC data review summaries, EPA SASD staff will make a final data usability determination of each data point for its coastal Alaska human health risk assessment.

Details regarding procedures, personnel, and quality control requirements needed for the activities summarized above project are documented in the remainder of this QAPP.

Table 1 List of Target Pollutants in Cook Inlet Contaminant Study
Organohalide pesticides
Dioxins/furans
Toxic PCBs
Hydrocarbons, Phenols, Polycyclic Aromatic Hydrocarbons and PAH Metabolites
Total mercury
Methyl mercury
Total arsenic
Inorganic arsenic
Arsenic (III)
Arsenic (V)
Monomethylarsonic acid (MMA)
Dimethylarsonic acid (DMA)
Chromium
Selenium
Cadmium
Lead

5.0 Quality Objectives and Criteria

As noted previously, this QAPP is applicable only to the sample analysis activities associated with the Cook Inlet Contaminant Study. Therefore, the quality objectives described in this section are primarily focused on those related to measurement aspects only, and are referred to as measurement quality objectives (MQOs). Data quality objectives associated with the overall study are addressed in the context of these MQOs and the DQOs cited in the "Combined Workplan and Quality Assurance Project Plan for the Cook Inlet Contaminant Study Sampling"(1).

MQOs for this project are described in terms of the following criteria: precision, accuracy, sensitivity, representativeness and completeness. Specific objectives for these criteria are given in Sections 5.1 through 5.4 below. Comparability is another frequently used criterion for measuring data quality. This criterion, however, reflects the overall sampling and analysis process; therefore, data quality objectives for this criterion is addressed the "Combined Workplan and Quality Assurance Project Plan for the Cook Inlet Contaminant Study Sampling" (1).

5.1 Precision

Precision is defined as the relative uncertainty about a given measurement and can be evaluated by comparing replicate sample results. In this study, overall analytical precision (i.e., precision associated with the sample homogenization, compositing, aliquotting, shipping, and analysis processes) will be assessed by evaluating results from duplicate sample aliquots prepared by the Sample Prep Lab and sent to each laboratory for analysis of the target pollutants. The Sample Prep Lab will prepare these duplicate aliquots at a frequency of 5% per matrix. The study MQO for overall precision is for results from 90% of these duplicate field sample pairs to produce results that agree within ± 30%.

Note: The design of this study includes a large number of other QC samples that provide information about the precision associated with various components of the analytical process. Details regarding these process-specific QC samples and specific MQOs for each are given in Section 12.

5.2 *Accuracy*

Accuracy is defined as the degree of agreement between an observed (e.g., measured) value and an accepted reference, or "true" value. In this study, overall accuracy of the analytical process will be measured by preparing and analyzing spiked field samples. Depending on the method used for analysis of individual pollutants targeted in this study, the spiked field samples may take the form of (1) matrix spike samples, which are field samples spiked with the analytes of interest at a concentration of approximately 1-5 times the background concentration or the ML, whichever is greater, (2) field samples spiked with isotopically labeled compounds, (3) field compounds spiked with surrogate compounds that are expected to behave in a manner similar to the target analytes but that are not expected to be present in the field samples collected, or (4) some combination of items 1 and 3.

The measurement quality objective for overall analytical accuracy in this study is for 90% of the spiked field sample results to fall within the acceptance criteria specified for each method listed in Table 2.

Table 2: Measurement Quality Objectives for Accuracy*

Analyte	Measure	Frequency	Method
Organohalide pesticides	Matrix spike and matrix spike duplicate (MS/MSD) samples	Spikes of all target analytes in 1 pair of samples per 10 samples of each matrix type	Modified Method 1656
	Surrogate compound spikes	At least one surrogate compounds spiked into every sample	
Dioxins/furans	Labeled compound spikes	Spikes of all target compounds in each sample	Method 1613, Revision B
Co-planar PCBs	Labeled compound spikes	Spikes of all target compounds in each sample	Method 1668
Hydrocarbons, Phenols, PAH/ PAHMetabolites	Labeled compound spikes	Spikes of all target compounds in each sample	GC/Selective detector
Total mercury	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Modified Method 1631
Methylmercury	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Research method
Total arsenic	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Modified Method 1638
Arsenic (III)	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Quartz furnace AA
Arsenic (V)	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Quartz furnace AA

Table 2: Measurement Quality Objectives for Accuracy*			
Analyte	Measure	Frequency	Method
MMA	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Quartz furnace AA
DMA	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Quartz furnace AA
Inorganic arsenic	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Quartz furnace AA
Total chromium	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Modified Method 1638
Seleneium	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Modified Method 1638
Cadmium	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Modified Method 1638
Lead	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Modified Method 1638

* Overall MQO for accuracy in study is for 90% of accuracy measurements to fall within the individual measurement MQOs listed in the table.

Note: The design of this study includes a large number of additional QC samples that provide information about the accuracy associated with various components of the analytical process. Details regarding these process-specific QC samples and specific MQOs for each are given in Section 12.

5.3 Sensitivity

Analytical sensitivity is defined as the minimum concentration of an analyte above which a data user can be confident that the analyte was reliably detected and quantified. For this study, the method detection limit (MDL) and the minimum level (ML) will be used to define the sensitivity of each measurement process.

The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero ; it is determined from analysis of a sample in a given matrix containing the analyte. The MDL results from estimating a method's sensitivity at the two lowest levels, zero concentration, and the lowest concentration that the method is capable of distinguishing from zero with a 99% probability. Procedures for determining an MDL are given at 40 *CFR* 136, Appendix B.

The ML is defined as the lowest concentration at which the entire analytical system must give a recognizable signal and acceptable calibration point for an analyte. It is equivalent to the concentration of the lowest calibration standard analyzed by a specific analytical procedure, assuming that all the method-specified sample weights, volumes, and processing steps have been employed.

It is not feasible to specify numeric objectives concerning the sensitivity of most pollutants targeted in this study because published methods do not exist for analysis of these pollutants in marine plant or tissue matrices. The following general DQOs have been developed based on risk assessment needs and existing measurement capabilities:

- State-of-the-art measurement techniques should be implemented for analysis of all target pollutants in order to obtain measurements that are as sensitive as are reasonably feasible.
- All laboratories must perform an MDL study to determine the MDL associated with each analyte they measure in the Cook Inlet Contaminant Study. The MDL for each target pollutant must be no more than one-third the concentration of that pollutant in the lowest concentration standard used by the laboratory to calibrate their instrument.
- If the method employed by the laboratory specifies an ML, the laboratory must be capable of meeting that ML, unless otherwise approved. If such deviations are approved, this QAPP will be amended accordingly.
- An MDL of 0.7 ppt is required for 2,3,7,8-TCDD and 2,3,7,8-TCDF.
- An ML of 0.2 ug/L is required for total arsenic.
- This is not a method development study; laboratories participating in this study will employ analtyical methods that they (or other researchers) have utilized previously in the performance of similar work. Method sensitivity will be dependent upon existing capabilities.

5.4 Completeness

Completeness is defined in terms of the percentage of data that are collected and deemed to be acceptable for use in this study. Three measures of completeness can be defined, as follows:

- Sampling Completeness, which is the number of valid samples collected relative to the number of samples planned for collection;
- Analytical Completeness, which is the number of valid sample measurements relative to the number of valid samples collected; and
- Overall Completeness, which is the number of valid sample measurements relative to the number of samples planned for collection.

Completeness goals are presented in Table 3.

Table 3
Measurement Quality Objectives for Completeness

MQO	Measure	Analyte	Acceptance Criteria
Sampling completeness	Number of valid samples collected relative to the number of samples planned for collection	Not Applicable	90% (see reference 1)
Analytical completeness	Number of valid sample measurements relative to the number of valid samples collected	All	90%
Overall completeness	Number of valid sample measurements relative to the number of samples planned for collection	All	81% (determined by multiplying sampling and analytical completeness goals).

5.5 Representativeness

Representativeness is an indication of (1) the degree to which a sample from a given site is typical of that site or area (e.g., a coastal harbor) and the matrix from which it was taken (e.g., a tissue matrix), and

(2) the degree to which the sample accounts for analyte heterogeneity in the matrix. Data quality objectives for the representativeness of the samples and matrices collected are discussed in the "Combined Workplan and Quality Assurance Project Plan for the Cook Inlet Contaminant Study Sampling" (June 5, 1997) and are not repeated here.

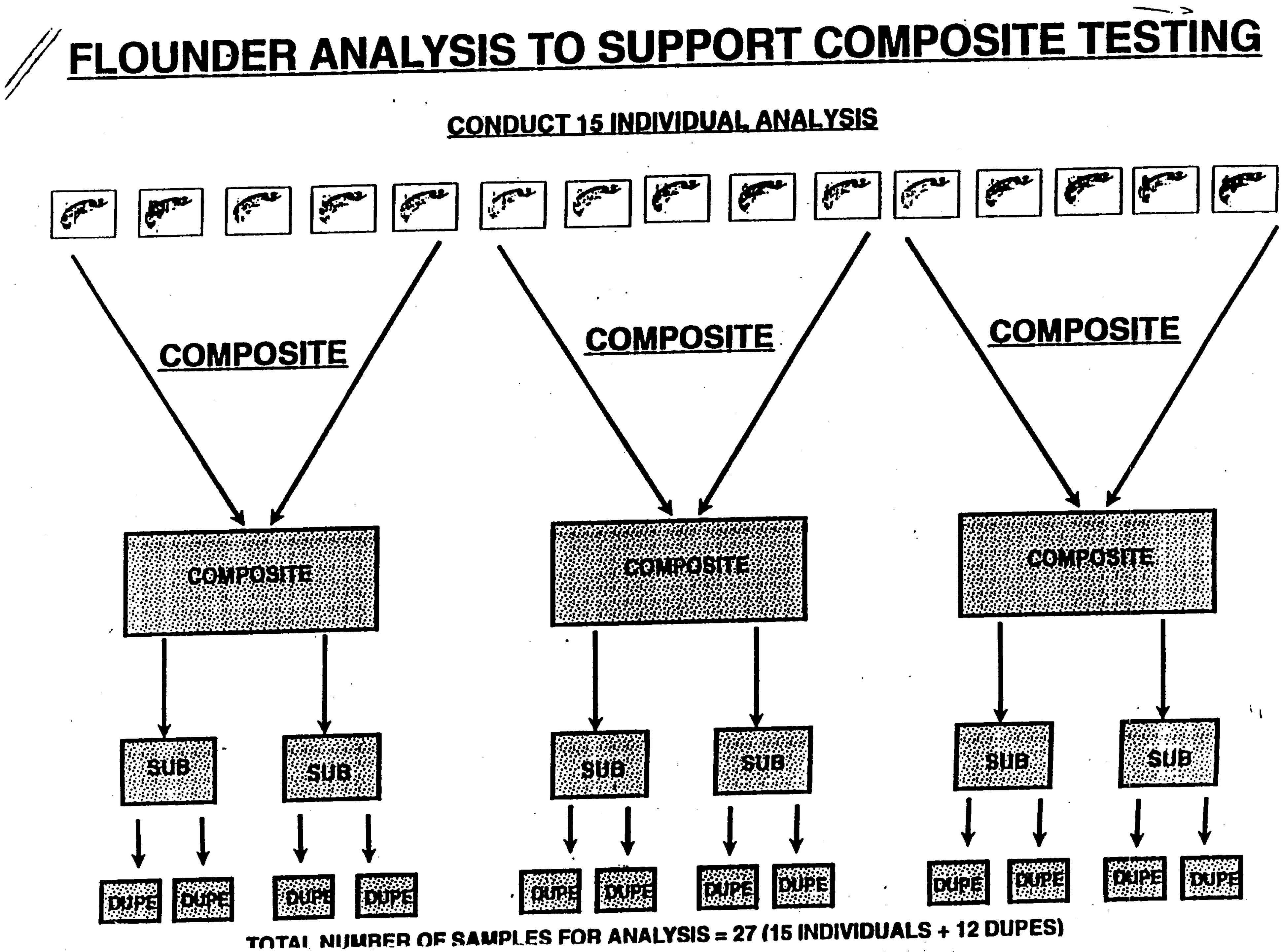
An in-depth study of sample homogeneity and the effectiveness of the compositing procedure will be performed with three sets of flounder samples collected from Port Graham Village. Flounder collected in these sets will be prepared as individuals and as composites, and the composite samples will be subsampled and replicated for subsequent analysis by the analytical laboratories. This representativeness study, which is illustrated in Figure 2, will be performed as follows.

- Three sets of five flounder samples, for a total of 15 individual flounder, will be collected from Port Graham Village and sent to the Sample Prep Laboratory.
- Upon receipt, the Sample Prep Lab will homogenize each flounder individually, and aliquot samples of these 15 individuals for each of the analyses in Table 1. These individual homogenates will be shipped to the analytical laboratories for analysis.
- Once the individual flounder homogenate samples have been created and measured out of their respective container for the analytical laboratories, the Sample Prep Lab will composite the remaining homogenate from eahc group of five individuals to generate three distinct composite samples.
- After these three composite samples have been created, the Sample Prep Lab will split each composite into two subsamples.
- The Sample Prep Lab will then split each of the two subsamples into two replicate samples (for a total of four replicate samples). The aliquots for the analytical laboratories are to be prepared from these four replicate samples. This will result in a total of 12 samples from the three original ocmposite samples. Each of these 12 replicate samples are to be given a distinct EPA sample number so that laboratories are not aware of their replicate nature.
- A total of 15 homogenates, each of which represents an individual flounder, and a total of 12 replicate samples that are the consequence of creating 4 aliquots from each of the 3 composite samples will have been prepared.

To quantify variability introduced during the sample compositing process, results of the replicated composite samples will be compared with results from the individual homogenates. The measurement quality objective for this test is to for the relative standard deviation of replicate composite results and mathematical composite results (calculated from results of individual sample analyses) to agree within ± 30%.

Because resource constraints make it impractical to perform such an in-depth representativeness study in all Cook Inlet matrices sampled, the Sample Prep Lab will prepare duplicate samples at a frequency of 5% (1 per 20) per matrix per method/analyte, assign unique EPA sample numbers to each of the duplicate samples, subdivide each duplicate into a sufficient number of aliquots necessary for analysis of all target pollutants in Table 1, and ship the aliquots to the designated analytical laboratories for analysis. The relative percent difference (RPD) between each duplicate sample pair will be evaluated to confirm that the sample aliquots prepared by the Sample Prep Lab are homogeneous, and therefore, representative of the sample matrix from which they were prepared. The measurement quality objective for these Sample Prep Lab duplicates is± 30%.

Figure 2



FLOUNDER ANALYSIS TO SUPPORT COMPOSITE TESTING
CONDUCT 15 INDIVIDUAL ANALYSIS
COMPOSITE
COMPOSITE
COMPOSITE
COMPOSITE
COMPOSITE
COMPOSITE
SUB
SUB
SUB
SUB
SUB
SUB
DUPE
DUPE
DUPE
DUPE
DUPE
DUPE
DUPE
DUPE
DUPE
DUPE
DUPE
DUPE
TOTAL NUMBER OF SAMPLES FOR ANALYSIS = 27 (15 INDIVIDUALS + 12 DUPES)

6.0 Project Narrative

As noted previously, data gathered during this study will be used to assess health risks to Native American tribes living in coastal Alaska. The success of the project's analytical phase will be determined by evaluating the extent to which the MQOs established in this QAPP are met. Because many of the sample preparation and analysis methods used in this study are research methods, another measure of project success will be the degree to which EPA is able to use the "lessons learned" in this project for the subsequent development of EPA methods applicable to commercial laboratory environments.

To ensure that project MQOs are met, EAD will:

- Identify the best analytical methods that meet overall DQOs for this project. Methods selected will be chosen based on (1) their ability to meet the detection and quantitation limits needed for human health risk assessments, (2) their applicability to the matrices and pollutants of interest in this study, and (3) recommendations from recognized experts in the fields of analytical and marine chemistry. Published or draft EPA methods applicable to commercial laboratory environments will be selected if they meet these criteria. If, however, existing EPA methods do not meet these criteria, EAD and SCC staff will review the public literature and solicit input from recognized experts to identify alternate methodologies.
- Require laboratories to perform the standard EPA "600/1600 series" QC requirements for each type of analysis. If the methods selected do not contain all of these QC requirements, EAD will supplement the methods with these requirements.
- Prepare detailed "Analytical Requirements Summaries" for the Sample Prep and the Sample Analysis phases of the project. These Analytical Requirements Summaries will describe the number and type of samples to be collected and analyzed, the sampling and analysis schedule, sample handling and analysis procedures, quality assurance (QA) and quality control (QC) procedures, and data reporting requirements. These Analytical Requirements Summaries will supplement existing EAD megalab contracts that already specify minimum technical, personnel, reporting, facility, and QA/QC requirements.
- Require laboratories to demonstrate that they are qualified to perform the required tasks by submitting data from similar analyses prior to commencement of the study. These data may be from similar studies, MDL studies, and/or IPR studies.
- Direct SCC to monitor laboratory performance throughout the study by maintaining routine communication with the laboratories and with EPA to identify and resolve any problems that arise.
- Direct SCC to carefully review each data submission to verify that the required methods were followed, and that all QC acceptance criteria and MQOs established for this study were met.

Following completion of its data quality assessments, SCC will document in writing its assessment of the data and forward these data review narratives to EPA for use in determining the acceptability of the data against overall DQOs established for the project.

7.0 Special Training Requirements

The EAD megalab contracts specify minimum skills required of the analysts that perform work under those contracts. Laboratory staff also must be experienced in using the procedures and instruments (e.g., GC/MS, CVAFS, etc) that will be required for this study.

SCC staff responsible for reviewing data must be experienced in performing data review activities, must have been trained to review data in accordance with EAD's general data review guidelines, must be experienced in reviewing data generated with the instrumentation that will be used in this study, and must be familiar with the DQOs established for this study. For example, any SCC staff member that reviews dioxins/furans data generated during this project must have some prior experience reviewing data generated by HRGC/HRMS, and must have been trained to review such data in accordance with EAD's general data review guidelines. This reviewer also must have read and understood the DQOs applicable to this study.

8.0 Documentation and Records

Documentation and records relating to the analytical phase of this project will include 1) written documentation of analytical requirements, 2) hardcopy and electronic records generated by the Sample Prep Lab and the analytical laboratories, and 3) records generated by SCC.

8.1 Documentation of Analytical Requirements

As noted previously, specific analytical requirements relating to sample preparation, storage, homogenization, compositing, shipment, and analysis will be documented in Analytical Requirements Summaries that will supplement the EPA "megalab" contracts. These Analytical Requirements Summaries also will detail Sample Prep Lab and analytical laboratory requirements for data documentation and reporting. In general, all laboratories will be required to meet the following minimum documentation and record keeping requirements:

- Submit summary reports of all analytical results. These summary reports must be provided in both hardcopy and electronic format.
- Submit copies of all raw data in hardcopy format. Raw data will include items such as quantitation reports, strip charts, spectra, bench sheets, and laboratory notebooks showing tare and sample weights, and sample volumes. Raw data also will include any other information that would allow an independent reviewer to verify the calculations performed and trace the final results to the raw data. Laboratories will be required to clearly identify each data element in their data package(s).
- Submit a written report that details any problems encountered during analysis of the samples. The written report also should include comments on the performance of any part of a method.
- Obtain pre-approval of any modifications to the analytical techniques specified and submit detailed explanations of the changes implemented.
- Report results consistently in the reporting units (e.g., ug/L, ng/L, etc) specified in the Analytical Requirements Summary of the contract delivery order issued for this project.

8.2 SCC Records

SCC will create and maintain a master study file, separate files for each sampling "episode", and data review files. For this study, an episode will be defined as a group of samples collected from a single village within a finite period of time. SCC will assign a unique four digit number to each episode; this episode number will be used by EPA, the sampling contractors, the laboratories, and SCC to reference the sampling event. In addition, SCC will provide the Sample Prep Lab with a series of five digit EPA sample numbers that will be assigned to each sample after compositing and aliquoting. These five digit EPA sample numbers will be used by the Sample Prep Lab, the analytical laboratories, SCC, and EPA to identify each sample analyzed during the study. This approach of using episode and sampling numbers to identify the sampling events and samples collected provides a means for tracking each sample without revealing the location from which it was collected. SCC also will provide the Sample Prep Lab with a list of analytical laboratories and EPA "traffic reports" for use in documenting and shipping samples to the analytical laboratories.

Cumulatively, the master file, the episode files, and the data review files will contain the following records:

- A copy of the QAPPs for the sampling and analysis phases of this project
- A copy of each Analytical Requirements Summary prepared for this project
- A summary page that documents the Episode Number, the sample numbers assigned to the Episode, the laboratories that will be analyzing each sample, the EPA contract number under which these analyses are being performed, and the date of sample collection and shipment
- The name, address, phone number and primary contact of each laboratory preparing and analyzing samples in the episode
- A copy of each traffic report prepared and sent with each sample
- A list that cross-references the number(s) assigned to each sample by the sample collection team against the five-digit EPA sample numbers assigned by the Sample Prep Lab after homogenization, compositing, and aliquotting
- A log of all verbal communication with laboratory staff, sampling personnel, and EPA staff regarding the status of or problems with the study
- Copies of all written correspondence with laboratory staff, sampling personnel, and EPA staff regarding the study
- Complete records regarding the data review process, including a final copy of SCC's written data review narrative and the final data submission from each laboratory
- A database of final analytical results associated with each field sample.

SCC will provide copies of the final laboratory submissions, copies of the final data review narratives, and a copy of the final database to EPA after the data reviews are complete. SCC will retain the master file, each episode file, and copies of each laboratory data submission. SCC will provide copies of these materials on an as-needed basis to EPA upon request.

9.0 Sampling Process Design and Sampling Methods

Whole fish, whole invertebrate, marine mammal organ, and marine plant samples will be collected from the Tyonex, Seldovia, Port Graham, and Nanwalek villages. Details regarding the number and types of samples to be collected, sampling locations and frequencies, and other aspects of the sampling design are documented in Section 2.1 of the "Combined Workplan and Quality Assurance Project Plan for the Cook Inlet Contaminant Study Sampling" (June 5, 1997) and are not repeated here. Similarly, details regarding sampling methods and equipment and field storage and shipping procedures are documented in Section 2.2 of that document and are not repeated here.

10.0 Sample Handling and Custody Requirements

Details regarding the handling and documentation of samples by sampling personnel are detailed in Section 2.3 of the "Combined Workplan and Quality Assurance Project Plan for the Cook Inlet Contaminant Study Sampling" (June 5, 1997) and are not repeated here. Instead, this QAPP details sample and data handling requirements for the Sample Prep Lab. the analytical laboratories, and SCC staff.

10.1 General EAD Sample Handling and Custody Requirements

Formal chain of custody procedures are not required and will not be used for this project. Instead, the laboratories will follow the general sample handling and documentation procedures that are routinely used by EAD. Standard EAD procedures are detailed in the EAD megalab contracts and include the following:

- Each sample sent to analytical laboratories for analysis will be identified with a unique, five-digit EPA sample number to be used by EPA, SCC, and laboratory staff for sample tracking purposes. Samples also will be grouped into "episodes", with each episode representing a group of samples collected from a single site (i.e., village) during a finite period of time. Each episode is assigned a unique four-digit number to facilitate sample tracking.
- Normally, field personnel assign EPA sample numbers, episode numbers, and traffic reports when collecting and shipping samples in the field. In this study, however, field personnel will use alternate sample handling and documentation procedures, as described in Sections 1.7, 2.2, and 2.3 of the "Combined Workplan and Quality Assurance Project Plan for the Cook Inlet Contaminant Study Sampling".(1) Therefore, the Sample Prep Lab will label each sample container with the appropriate EPA sample number and document the sample number and the associated episode number on an EPA traffic report after preparing (e.g., compositing, homogenizing, and aliquotting) the samples.
- SCC staff will provide the five-digit EPA sample numbers, instructions for sample numbering, and EPA traffic reports to the Sample Prep Lab. The Sample Prep Lab will assign a unique EPA sample number to each composite sample and each individual sample scheduled for analysis. The Sample Prep Lab also will assign an EPA sample number to any laboratory QC sample it has prepared for subsequent analysis by the analytical laboratories. The Sample Prep Lab will be responsible for affixing sample labels to the sample containers that are shipped to the analytical laboratories and for documenting sample numbers and shipping information on each traffic report.
- Immediately after shipping samples, the Sample Prep Lab will notify SCC of the number of samples shipped, the airbill tracking number(s), the designated recipient(s), and the anticipated arrival date. The Sample Prep Lab will retain one copy of the EPA traffic report and forward one copy to SCC. The remaining two copies will be shipped along with the samples to the analytical laboratories. *Note*: The field sampling team also will be responsible for communicating sample shipment information to SCC and for providing SCC with copies of documentation prepared in the field.
- When shipping samples, field sampling personnel and Sample Prep Lab personnel will ensure that samples are packaged at appropriate temperatures and in such a way as to prevent breakage of bottles. They also will ship samples in accordance with applicable federal, state, and local regulations.
- After receiving shipping information from the field sampling teams or Sample Prep Lab, SCC will document the information in the episode file and notify the recipient laboratory of the anticipated shipment. On the day that samples are scheduled to arrive, SCC will again contact the laboratory confirm that samples arrived on schedule and in good condition.
- In the event that samples are lost or damaged in transit, SCC will work with the laboratories, EPA, field personnel, and/or the shipping carrier to resolve the problem. SCC will document any such problems and resolutions in the episode file.
- Following receipt of samples at the analytical laboratories, SCC staff will maintain communication

with laboratory personnel throughout the duration of analysis. The purpose of this communication is to promptly identify, resolve, and document any problems that arise during the course of analysis.

- Upon receipt of samples from the Sample Prep Lab, the analytical laboratories will sign and date the EPA traffic reports, retain one copy and return one copy to SCC to document the date and condition of samples upon receipt.

10.2 Additional Sample Handling and Custody Requirements Applicable to this Study

Additional procedures that will be implemented for this study will be detailed in the Analytical Requirements Summaries prepared to supplement the "megalab" contracts and in the analytical methods specified in the Analytical Requirements Summaries. These additional procedures will include the following:

- *Storage:* Each laboratory will be required to store all samples and sample aliquots at -20° C.
- *Shipping*: When shipping samples to the analytical laboratories, the Sample Prep Lab must pack the samples with enough dry ice to maintain a cooler temperature of -4° C.
- *Handling:* As necessary to preclude contamination during the sample handling and analysis processes, each laboratory will be required to use "clean" protocols appropriate for the target analytes and sensitivity required in this study. The Sample Prep Laboratory also will be required to rinse samples with distilled water prior to homogenization and compositing.
- *Batch Compositing Procedure*: Unless otherwise noted by SCC or the field sampling team, all samples requiring compositing (i.e., all but the octopus and marine mammal organ samples) will be composited using the "batch" method, in which all of the individual specimens that comprise the sample are homogenized together, regardless of each individual's proportion to one another (as opposed to the "individual" method, in which equal weights of each specimen are added together).
- *Sample Numbering:* Unless otherwise noted by SCC or the field sampling team, the Sample Prep Lab will assign a unique EPA sample number to each composite sample, and will assign the same EPA sample number to each aliquot of a single composite. Similarly, the Sample Prep Lab will assign a unique EPA sample number to each individual octopus or marine mammal organ sample, and will assign the same EPA sample number to each aliquot taken from that sample after homogenization.
- *Fish Preparation:* When homogenizing fish samples, the Sample Prep Lab will homogenize all samples as thoroughly as possible, including all parts of the fish. The Sample Prep Lab will take care to ensure that no chunks of skin or tissue remain. With the exception of 15 flounder from Port Graham Village (see "Evaluating Compositing Variability" below) fish samples will be homogenized in the composite groups identified by the field sampling personnel, and after homogenization, the Sample Prep Lab will assign a single five-digit EPA number to each homogenized composite group, as instructed by SCC. Following assignment of the EPA sample number to each composite, the Sample Prep Lab will subdivide each fish composite sample into replicate aliquots as necessary for subsequent analysis. The Sample Prep Lab will store each aliquot in a contaminant-free container and label each container with the sample number corresponding to that composite.
- *Plant Preparation:* Plant samples will have been collected and stored by the field sampling personnel in 1-L glass jars as composites. Upon receipt of these composite plant samples, the Sample Prep Lab will homogenize the contents of each jar, assign a unique five-digit EPA sample number to each composite as instructed by SCC, and subdivide each composite into appropriate aliquots as necessary for subsequent analysis. The Sample Prep Lab will store each aliquot in a

contaminant-free container and label each container with the sample number corresponding to that composite.

- *Marine Invertebrate Preparation:* For marine invertebrate samples of clams, mussels, chitons, and snails, the Sample Prep Lab will remove all tissue material from the shell of each specimen prior to homogenization. The Sample Prep Lab will homogenize the entire tissue contents of these invertebrate samples in the composite groups identified by the field sampling personnel, assign a unique five-digit EPA sample number to each composite as instructed by SCC, and subdivide each composite into appropriate aliquots as necessary for subsequent analysis. The Sample Prep Lab will store each aliquot in a contaminant-free container and label each container with the sample number corresponding to that composite.
- *Mammal Organ and Octopi Preparation:* Marine mammal organ and octopi samples will be analyzed as individual samples rather than composites. Therefore, the Sample Prep Lab will homogenize each entire animal (for octopi) or each entire organ (for mammals) separately (compositing is not required), and subdivide each homogenized specimen into replicate aliquots as necessary for subsequent analysis. The Sample Prep Lab will store individual aliquots of each mammal organ or octopus sample in contaminant-free containers and label each container with the unique five-digit EPA sample number sample number corresponding to that sample.
- *Extra Sample Volume*: If enough sample is available, the Sample Prep Laboratory will aliquot and store an additional 400 grams of each sample so that additional homogenate is available in case of problems that arise during shipment or analysis of samples.
- *Evaluating Compositing Variability*: In order to measure potential variability of the compositing procedure, the Sample Prep Lab will prepare individual and composite samples for three sets of flounder samples from the Port Graham Village. Each of these sets will consist of five individuals, for a total of 15 flounder. The laboratory will homogenize each fish individually and prepare, package, and ship aliquots in contaminant-free containers to the analytical laboratories for analysis of the pollutants listed in Table 1. The Sample Prep Lab also will composite the remaining homogenate from each group of five individuals to generate three distinct composite samples. Once these three composites have been created, the Sample Prep Lab will split each composite into two subsamples, and further split each subsample into two replicates (for a total of four replicate subsamples from each composite). This will result in a total of 12 samples from the 3 original composites. The Sample Prep Lab will then subdivide each of these 12 replicates into individual aliquots for subsequent of the pollutants listed in Table 1
- *Verification of Homogeneity*: To further ensure that individual aliquots are homogeneous, the Sample Prep Lab will prepare duplicate samples at a frequency of one in 20 samples per analyte group or method. These duplicate samples will be assigned an EPA sample number that is different from the sample number assigned to the original homogenized sample.
- *Trip Blanks*: To ensure that the coolers used for shipment of the homogenized aliquots to the analytical laboratories are free of contamination, and to assess any cross-over contamination, the Sample Prep Lab will prepare trip blank samples. The Sample Prep Lab will store and ship these samples in the same type of containers used for storage and shipment of the field sample aliquots.

11.0 Analytical Methods Requirements

Samples collected under this study will be analyzed for organochlorine pesticides, dioxins/furans, co-planar PCBs, hydrocarbons, phenols, polycyclic aromatic hydrocarbons (PAHs) and PAH metabolites, total mercury, methyl mercury, total arsenic, arsenic (III), arsenic (V), monomethylarsonic acid, dimethylarsonic acid, inorganic arsenic,, total chromium, selenium, cadmium, and lead. These pollutants are listed in Table 1.

Published EPA methods do not exist for analysis of all the target pollutants in the marine plant and tissue matrices to be sampled in this study. Therefore, the following approach will be taken to identify appropriate methodology:

- If feasible, existing EPA methods will be modified to accomodate the matrices or pollants of interest. In such cases, modifications will be thoroughly documented by EPA in an Analytical Requirements Summary issued as part of a delivery order under a megalab contract.
- If existing EPA methods cannot be identified, environmental analysis methods published by other organizations, such as the American Society of Test Materials (ASTM) and the Food and Drug Administration (FDA), will be evaluated. Where appropriate, these methods will be specified for use as written or with modifications. These specifications will be documented by EPA in an Analytical Requirements Summary issued as part of a delivery order under a megalab contract.
- If published methods are not available at all, methodologies described in public literature will be evaluated. Where appropriate, these techniques will be documented by EPA in an Analytical Requirements Summary issued as part of a delivery order under a megalab contract.
- If no methods have been published by EPA or other organizations, and if no clearly suitable methods can be identified in the public literature, EPA will specify general analytical needs, in terms of matrices, target pollutants, and required sensitivity, in an Analytical Requirements Summary issued as part of a delivery order under a megalab contract. EPA also will request that laboratories submitting bids to perform work under this delivery order also submit recommendations concerning appropriate methodology, the rationale for selecting this methodology, and sensitivity and performance criteria that might be expected when using this methodology on the matrices being sampled in this study. EPA will select a technique after evaluating the recommendations selected.

Sections 11.1 through 11.8 below summarize the methodologies that EPA expects to use in the Cook Inlet Contaminant Study. It should be noted that alternate techniques may be considered and implemented based on further discussions with experts in the field of environmental chemistry. In such cases, these alternate techniques will be documented in the Analytical Requirements Summaries issued by EPA.

11.1 Organohalide Pesticides and PCBs

Organohalide pesticides and polychlorinated biphenyls (PCBs) will be analyzed by Method 1656 (*Determination of Organo-halide Pesticides in Municipal and Industrial Wastewater*) with modifications designed to allow for extraction of the target pollutants from tissue and plant samples. Method 1656 was published in *Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater*, Revision 1, August 1993, EPA 821-R-93-010A). A list of the organohalide pesticides and PCBs that will be targeted in this study is provided in Appendix A of this QAPP.

Method 1656 is applicable to determination of a large number of organo-halide pesticides and certain PCBs (as Aroclors) in aqueous, solid, and semi-solid matrices. Modifications will be necessary to adapt the method to marine tissue and plant analyses. For plant samples, the laboratory has the option of using either a Soxhlet extraction with a 1:1 hexane/acetone solution, or sonication, as described in Method 1656 for solid samples. The laboratory also may find that sodium sulfate is not needed during the extraction process. the The laboratory will be provided the option of omitting this step from the procedure provided it can demonstrate that this ommission does not adversely affect the analysis. For tissue samples, the laboratory will fortify a 30 gram aliquot with surrogate compounds, grind the sample with 60 grams of anhydrous sodium sulfate, and blend it with 200 mL of petroleum ether solvent. For PCB determination, the laboratory also will clean up the extract using sulfuric acid.

Following sample extraction, both the plant and tissue extracts will be dried over sodium sulfate, concentrated using a Kuderna-Danish evaporator, cleaned up (if necessary) using gel permeation chromatography, adsorption chromatography, and/or solid-phase extraction, and then concentrated to 1 mL. After these extract concentration and cleanup steps have been performed, the laboratory will inject a 1 uL aliquot of the extract into the gas chromatograph (GC), where the organohalide pesticides will be separated on a wide-bore, fused-silica capillary column. The analytes can then be detected by an electron capture, microcoulometric, or electrolytic conductivity detector.

Qualitative analysis (e.g., identification of each pollutant) will be performed by comparing the GC retention times of each pollutant on two different columns with the respective retention times of an authentic standard. Quantitative analysis (e.g., determining the concentration of each pollutant) will be performed using an authentic standard to produce a calibration factor or calibration curve, and using the calibration data to determine the concentration of a pollutant in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume. Quality is assured through reproducible calibration and testing of the extraction and GC systems.

In order to further evaluate the quality of data generated using the modified method, the analytical laboratory will be required to use the modified method to perform an MDL study in a corn oil matrix and will be required to prepare IPR samples (in reagent water) that are spiked with all the target analytes listed in Appendix A. The laboratory also will be required to use corn oil as the reference matrix for animal tissue samples and reagent water as the reference matrix for plant and aqueous samples.

Finally, the megalab contractors will be provided with an opportunity to propose an alternate procedure either the extraction of tissue and plant samples and/or analysis by Method 1656. If the laboratories choose to propose alternate technologies, they will be requried to submit a summary of the procedure being proposed, QC elements and acceptance criteria that are believed to be appropriate for the proposed procedure, and a rationale for the suggested performance criteria.

11.2 Dioxins/Furans

Dioxins/furans will be analyzed by Revision B of EPA Method 1613 (*Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS*, October 1994, EPA821-B-94-005). Method 1613 is applicable to determination of tetra-through octa- chlorinated dibenzo-*p*-dioxins (CDDs) and dibenzofurans (CDFs) in water, soil, sediment, sludge, tissue, and other sample matrices. All seventeen 2,3,7,8-substituted CDDs and CDFs listed in Table 1 of Method 1613 will be targeted in this study.

Method 1613 contains three extraction procedures that are applicable to this study. For solid samples, labeled analogs of the target compounds are spiked into a 10 g (dry weight) aliquot of the homogenized solid sample. The homogenized sample is then extracted in a Soxhlet-Dean Stark (SDS) extractor. It is anticipated that marine plant samples can be extracted using the above-described procedure. In the event that alternate procedures may yield improved results, however, laboratories will be provided an opportunity to document and implement alternate techniques. Tissue samples are extracted by either a hydrochloric acid (HCl) digestion or an SDS or Soxhlet extraction. In either case, a 20 g aliquot of the sample is homogenized, and a 10 g aliquot is placed in a bottle and spiked with labeled analogs of the target compounds. If the HCl digestion is used, a solution containing 200 mL of HCl and 200 mL of a methylene chlorine:hexane are added after equilibration, and the bottle is agitated for 12-24 hours. The extract is evaporated to dryness, and the lipid content is determined. If the SDS or Soxhlet extraction is used, the spiked 10 g aliquot is mixed with sodium sulfate, allowed to dry for 12 - 24 hours, and extracted for 18 - 24 hours using a 1:1 solution of methylene chloride and hexane in a Soxhlet extractor. The extract is evaporated to dryness, and the lipid content is determined.

After extraction of plant and tissue samples, $^{37}Cl_4$-labeled 2,3,7,8-TCDD is added to each extract to measure the efficiency of the cleanup process. Sample cleanups may include back-extraction with acid and/or base, and gel permeation, alumina, silica gel, Florisil, and activated carbon chromatography. High performance liquid chromatography (HPLC) can be used for further isolation of the 2,3,7,8-isomers or other specific isomers or congeners. Prior to the cleanup procedures cited above, tissue extracts are cleaned up using an anthropogenic isolation column, a batch silica gel adsorption, or sulfuric acid and base back-extraction, depending on the tissue extraction procedure used.

After cleanup, the extract is concentrated to near dryness. Immediately prior to injection, internal standards are added to each extract, and an aliquot of the extract is injected into the gas chromatograph (GC). The analytes are separated by the GC and detected by a high resolution mass spectrometer. Two exact m/z's are monitored for each analyte.

An individual CDD/CDF is identified by comparing the GC retention time and ion-abundance ratio of two exact m/z's with the corresponding retention time of an authentic standard and the theoretical or acquired ion-abundance ratio of the two exact m/z's. Isomer specificity for 2,3,7,8-TCDD and 2,3,7,8-TCDF is achieved using GC columns that resolve these isomers from other tetra-isomers.

Quantitative analysis for the seventeen target CDDs/CDFs and for the labeled compounds is performed using selected ion current profile (SICP) areas in one of two ways:

(1) For the fifteen 2,3,7,8-substituted CDDs/CDFs with labeled analogs, the GC/MS system is calibrated, and the concentration of each compound is determined using the isotope dilution technique

(2) For remaining two target compounds (1,2,3,7,8,9-HxCDD and OCDF) and for the labeled compounds, the GC/MS system is calibrated, and the concentration of each compound is determined using the internal standard technique.

The quality of the analysis is assured through reproducible calibration and testing of the extraction, cleanup, and GC/MS.

11.3 Toxic PCBs

Toxic PCBs will be analyzed by draft EPA Method 1668 (*Toxic Polychlorinated BiPhenyls by Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry* March 1997, EPA-821-R-97-001). The toxic PCBs listed in Table 1 of Method 1668 are to be targeted in this study. If feasible, full-PCB congener determinations also may be performed.

Method 1668 was based on and is procedurally similar to Method 1613. Differences in the method relate primarily to analytical columns used to separate the target compounds and extraction/cleanup procedures that are applicable to PCB determination. Method 1668 is applicable to determination of toxic PCBs in water, soil, sediment, sludge, tissue and other samples. The detection limits and quantitation limits in the method are usually dependent on the level of interferences rather than instrumental limitations. Table 2 of the method presents MLs that can be achieved when common laboratory interferences are present.

If full PCB congener analysis is determined to be feasible, the analytical laboratory will be required modify the method as necessary to determine the identity and concentration of each PCB congener by single-point calibration, internal standard, and HRGC/HRMS in the GC run on the SPB-octyl column in which the toxic congeners are determined. For congener pairs that are not separated, the laboratory will be required to employ an averaged response factor to estimate the concentration of the pair. The laboratory also will be required to perform a single-point calibration each time a five-point calibration for the toxic PCBs is performed. The laboratory will monitor two m/z's for each congene; the retention time and the ratio between the height or area of the signal at the two m/z's will be used for congener identification, and the area at the m/z's used for identification will be used for quantitation in the same way that these areas are used for quantitation of the toxic PCB congeners. The laboratory also will be required provide a report in the data package that lists the identity and concentration of the individual PCB congeners.

The quality of all analyses will be assured through reproducible calibration and testing of the extraction, cleanup, and GC/MS. In order to fully evaluate the quality of data generated using the method, the analytical laboratory will be required to use the method (with any approved modifications) to perform an MDL study in a corn oil matrix. The laboratory also will be required to prepare IPR samples (in reagent water) that are spiked with all the target analytes. The laboratory also will be required to use corn oil as the reference matrix for animal tissue samples and reagent water as the reference matrix for plant and aqueous samples.

11.4 Hydrocarbons, Phenols, Polycyclic Aromatic Hydrocarbons and PAH Metabolites

Because no established and approved procedure exists for measuring a broad range of the above-listed groups of compounds in tissue and plant matrices, laboratories will be asked to submit a proposal for analysis of these samples. Laboratories will be asked to prepare this proposal with the understanding that the focus of the study is to gather information to perform a human health risk assessment of native tribes in Alaska. As part of their proposals, laboratories will be asked to submit the following information to EPA:

1) The cost of the project itemized in per-sample units.
2) A summary of the procedure being proposed.
3) A target analyte list.
4) Suggested performance criteria for all matrices (i.e., animal tissue, plant, and water, at a minimum).
5) The rationale for determining these performance criteria.
6) Historical performance evaluation results (or equivalent QC analysis results), if available, to demonstrate that the method proposed is adequate and applicable to the matrices being analyzed.

EPA will evaluate the proposals submitted and select a technique that best reflect the objectives, schedule, and cost constraints associated with the Cook Inlet Contaminant Study.

In order to ensure that results using the approved technique are precise, accurate, and reproducible, EAD will require that all 600/1600 series QC requirements described in Section 12 of this QAPP be followed during analysis of study samples for hydrocarbons, phenols, PAHs, and PAH metabolites. These requirements are given in the "megalab" contracts and will be modified through the Analytical Requirements Summary as necessary for application to this study.

Prior to analyzing any field samples with the approved methodology, the laboratory will be required to perform an MDL study in a corn oil matrix. The laboratory also will be required to perform four IPR analyses in a reagent water matrix. The MDL and IPR tests must include all of the targeted hydrocarbon, phenol, PAH, and PAH metabolite compounds.

11.5 Total Mercury

Total mercury (Hg) will be analyzed by a modified version of draft EPA Method 1631 (*Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry*, July 1996, *get citation for method*). Method 1631 is designed for measurement of total Hg in filtered and unfiltered water in the range of 0.2 - 100 ng/L.

As written in the draft version of the method, a 100 - 2000 mL sample is collected into a specially cleaned, pretested, fluoropolymer bottle using sample handling techniques specially designed for collection of mercury at trace levels (3). The sample is either field- or laboratory- preserved by the addition of 5 mL of pretested 12 N HCl per liter of sample, depending on the time between sample collection and arrival at the laboratory. Sample preparation and analysis are conducted in laboratory facilities specially designed for determination of mercury at 0.2 - 100 ng/L concentration. At this facility, a 100 mL sample aliquot is placed in a specially designed purge vessel. Before analysis, 0.2 N BrCl is added to oxidize all Hg compounds to Hg(II). After oxidation, the sample is sequentially pre-reduced with $NH_2OH \cdot HCl$ to destroy the free halogens, and then reduced with $SnCl_2$ to convert Hg(II) to Hg(0). The Hg(0) is separated from solution by purging with nitrogen onto a gold-coated sand trap. The trapped Hg is thermally desorbed for the gold trap into an inert gas stream that carries the released Hg(0) into the cell of a cold-vapor atomic fluorescence spectrometer (CVAFS) for detection. Quality is ensured through calibration and testing of the oxidation, purging, and detection systems.

In this study, the tissue and plant samples will preserved by freezing the samples immediately after sample collection. HCl will not be used as a preserving agent. Method 1631 will be further modified by using pure HNO_3 for digestion of tissue samples prior to analysis. This tissue digestion procedure is described in *NOAA Technical Memorandum NOS ORCA 71*: "Sampling and Analytical Methods of the National Status and Trends Program National Benthic Surveillance and Mussel Watch Projects 1984 - 1992"(4).

It is anticipated that marine plant and tissue samples can be analyzed using the procedures described above. If, however, additional modifications to Method 1631 are believed to yield improved results, laboratories will be provided an opportunity to document and implement these alternate techniques.

11.6 Methyl Mercury

EPA procedures for the analysis of methyl mercury have not yet been published. Therefore, methyl mercury will be determined in this study by using a procedure developed and published by Dr.

Nicholas Bloom in the *Canadian Journal of Fish and Aquatic Science* (5).

In order to ensure that results using this research method are precise, accurate, and reproducible, EAD will require that all 600/1600 series QC requirements described in Section 12 of this QAPP be followed during analysis of samples in this study. These requirements are given in the "megalab" contracts and will be modified as necessary for application to this method in the Analytical Requirements Summary prepared for mercury analyses this study.

11.7 Total Arsenic, Selenium, Cadmium, and Lead

Total arsenic (As), selenium (Se), cadmium (Cd), and lead (Pb) will be determined in this study by a modified version of draft EPA Method 1638 (*Determination of Trace Elements in Ambient Waters by Inductively Coupled Plasma-Mass Spectrometry*, January 1996 Draft, EPA 821-R-96-005). Method 1638 was developed for determination of dissolved and total recoverable metals in ambient water. In this method, the samples are digested by gentle refluxing with nitric and hydrochloric acids. After cooling, the sample is made to volume, mixed, and centrifuged or allowed to settle overnight prior to analysis. In this study, the method will be modified to employ a nitric acid digestion procedure for digestion of the sample matrices collected in this study.

Following digestion, the sample will be introduced into a radio frequency plasma where energy transfer processes cause desolvation, atomization, and ionization. There, the ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio (m/z) by a mass spectrometer having a minimum resolution capability of 1 amu peak width at 5% peak height at m/z 300. Ions transmitted through the mass analyzer are detected by an electron multiplier or Faraday detector and the resulting current is processed by a data handling system. Quality will be assured through calibration and testing of the digestion and detection systems.

The quality of all analyses will be assured through reproducible calibration and testing of the digestion and ICP/MS systems. The laboratory also will be required to determine the MDL for each target metal prior to analysis of field samples. The laboratory will be required to use corn oil as the sample matrix in the MDL studies, provided that a background analysis shows no target analytes to be present in the corn oil. If metals are present in the corn oil, the MDL will be determined in reagent water. Similarly, OPR samples will be prepared in corn oil if no metals are detected in the background analysis. IPR samples will prepared in reagent water.

11.8 Speciation of Arsenic

In addition to total arsenic as described above, this study requires determination of arsenic (III), arsenic (V), monomethylarsonic acid (MMA), dimethylarsonic acid (DMA), and inorganic arsenic. Because an EPA method for determination of these forms of arsenic has not yet been published, a research method developed by Battelle Laboratories will be used to determine these pollutants in this study.(6)

In this technique, arsenic (III), arsenic (V), MMA, and DMA are volatilized from solution at a specific pH after reduction to the corresponding arsines with sodium borohydride. The volatilized arsines are then swept onto a liquid nitrogen cooled chromatographic trap, which upon warming, allows for a separation of species based on boiling points. The released arsines are swept by helium carrier gas into a quartz cuvette burner cell, where they are decomposed to atomic arsenic. Arsenic concentrations are determined by atomic absorption spectroscopy. Strictly speaking, this technique determines the valence states of arsenate (V) and arsenite (III) rather than the species of inorganic arsenic. The actual species of inorganic arsenic are assumed to be those predicted by a geochemical equilibrium model.

In order to ensure that results using this research method are precise, accurate, and reproducible, EAD will require that all 600/1600 series QC requirements described in Section 12 of this QAPP be followed during analysis of samples in this study. These requirements are given in the "megalab" contracts and will be modified as necessary for application to this method in the Analytical Requirements Summary prepared for determination of arsenic species.

As part of these QC requirements, the laboratory will be required to determine the MDL for each target metal prior to analysis of field samples. The laboratory will use corn oil as the sample matrix in the MDL studies, provided that a background analysis shows no target analytes to be present in the corn oil. If metals are present in the corn oil, the MDL will be determined in reagent water. Similarly, OPR samples will be prepared in corn oil if no metals are detected in the background analysis. IPR samples will prepared in reagent water.

12.0 Quality Control Requirements

All field sample analyses performed in this study will be performed in conjunction with the standard QC elements described in the EPA 600 and 1600 series methods. In developing these methods, EPA sought scientific and technical adv ice from many sources, including EPA's Science Advisory Board, scientists at EPA's environmental research laboratories, scientists in industry and academia, and scientists, managers and legal staff at EPA Headquarters. The result of discussions held among these groups was the standardized QA/QC approach that is an integral part of the 600- and 1600- series methods. Over the years, these QA/QC requirements have been refined to reflect improvements in environmental science and policy. The most current versions of the 600- and 1600- series QA/QC contains the following elements:

- Use of pure and traceable reference standards and periodic analysis of reference samples
- Demonstration of instrument calibration and system performance
- Calibration verification
- Absolute and relative retention time precision (for chromatographic analyses)
- Verification that the laboratory can achieve required MDLs and MLs
- Analysis of Initial Precision and Recovery (IPR) samples to demonstrate the laboratory can achieve precise and accurate results with the method prior to use on field samples
- Analysis of blanks to demonstrate freedom from contamination
- Recovery of surrogate or labeled compounds spiked into the sample to assess the effect of matrix interferences on compound identification and quantitation
- Matrix spike and matrix spike duplicate (MS/MSD) analyses to assess the effect of matrix interferences in non-isotope dilution methods
- Analysis of Ongoing Precision and Recovery (OPR) samples to demonstrate continued laboratory performance with the method
- Calculation of laboratory statements of data quality.

All of the EPA methods to be used in this study contain procedures for calculating each of the required QC statistics. The research methods to be used in this study, however, contain procedures for performing and calculating some, but not all, of the required QC statistics. In cases where the research method is silent on a required QC element, the procedures given in the *Guide to Method Flexibility and Approval of EPA Water Methods* will be followed. (7)

13.0 Instrument/Equipment Testing, Inspection, and Maintenance Requirements

Each laboratory participating in this study will be responsible for testing and inspecting the equipment used in this study. These laboratories also will be responsible for implementing preventative and corrective maintenance necessary to produce precise and accurate data that meets the measurement quality objectives listed in this QAPP. Specific requirements for maintaining the equipment at each laboratory will be documented in the laboratory QA plans used during the course of this study. Specific records of preventative maintenance, problems, and corrective actions will be documented by each laboratory in instrument log-books maintained on-site in the laboratory. These logbooks will be periodically reviewed by a laboratory manager/supervisor and will be available to an external audit team upon request.

14.0 Instrument Calibration and Frequency

All laboratories supporting this study will be required to calibrate instruments used in the study prior to analysis of field samples and to periodically verify calibration during the course of the study. Calibration standards used by the laboratories will need to be certified as to purity, concentration, and authenticity, or prepared from materials of known purity and composition. Detailed instrument calibration procedures will be specified in each of the analytical methods, the laboratory contracts, or the Analytical Requirements Summaries prepared for this study and are summarized as follows.

All methods employed in this study require a multi-point calibration prior to use of the instrument for analysis of field and QC samples. The frequency of this initial, multi-point calibration varies across methods due to variations in instrument stability and calibration procedures. While some methods, such as those that employ atomic absorption (AA) spectroscopy, require daily calibration of the instrument, other methods, such as those that employ GC/MS, require the instrument to be calibrated only if the calibration verification standard fails to meet pre-defined acceptance criteria. All methods require the laboratory to verify instrument calibration at least once per working shift during which samples are analyzed. Because the stability of instruments vary by instrument type, the length of an allowable working shift varies and is specified in each method.

15.0 Inspection/Acceptance Requirements for Supplies and Consumables

The analytical laboratories participating in this study will not be providing supplies or consumables to EPA.

16.0 Requirements for Acquisition of Non-direct Measurement Data

The analytical phase of this study will not involve the collection of data obtained from non-measurement sources such as computer databases, spreadsheets and programs, and literature files. The risk assessment phase of this study will rely on the analytical data collected during this study and on reference values and non-direct measurement data, such as dietary uptake values, reference toxicity values, exposure pathways, etc. All non-direct measurement data used by risk assessment staff will be obtained from peer reviewed literature or databases.

17.0 Data Management

17.1 Field Data Management

Procedures concerning the management of field sampling data are described in the "Combined Workplan and Quality Assurance Project Plan for the Cook Inlet Contaminant Study" (June 5, 1997) and are not repeated here.

17.2 Laboratory Data Management

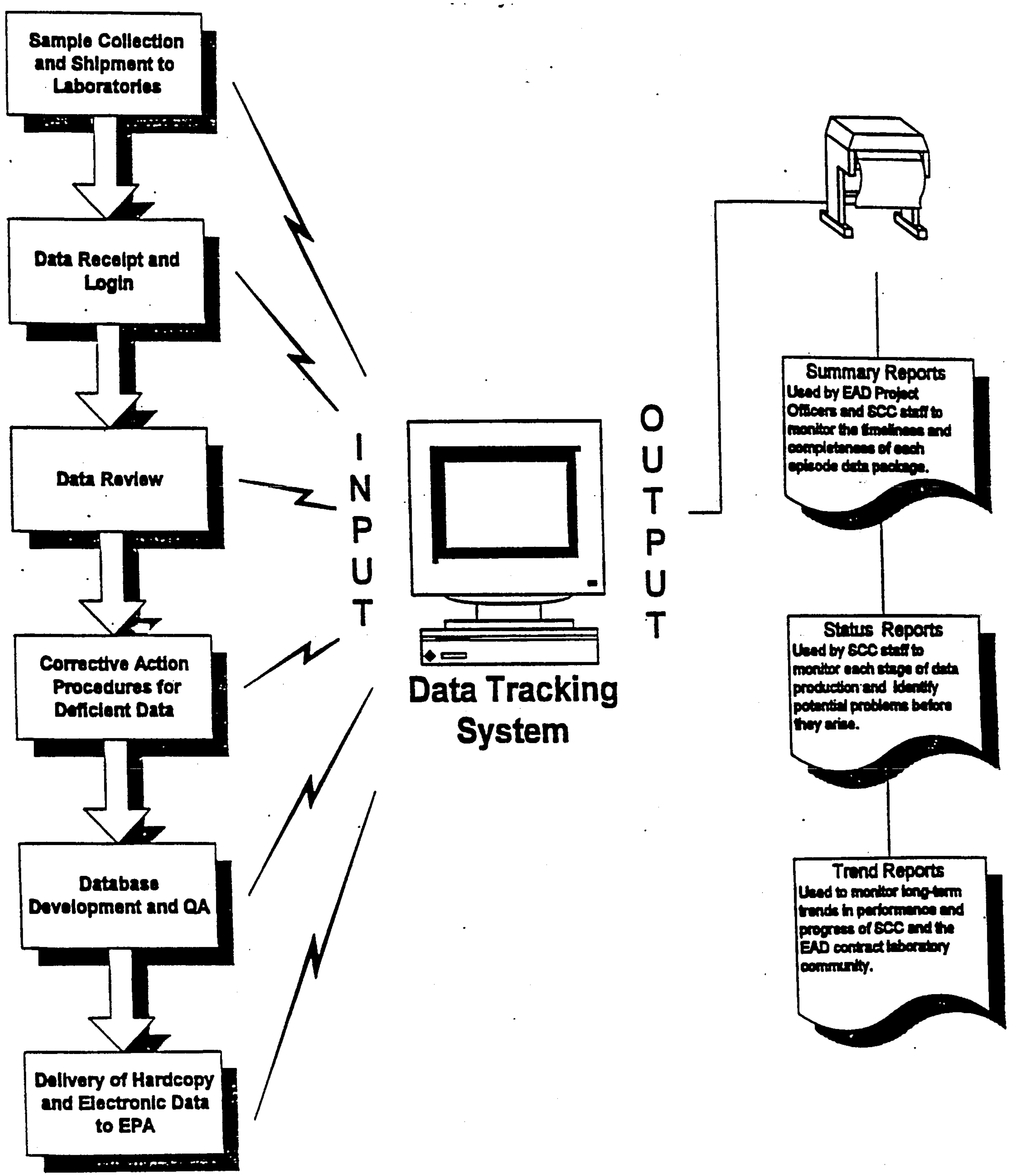
Laboratory data management procedures are detailed in the megalab contracts, but can be summarized as follows. Each laboratory participating in this study will be required to maintain all records and documentation associated with the preparation and analysis of Cook Inlet Contaminant Study samples for a minimum period of three years. To facilitate data tracking, each laboratory will be required to utilize EPA-assigned episode and sample numbers when reporting results. All results of sample analyses, labeled and native standards, surrogate compounds, spike compounds, and blanks must be reported on hardcopy and on magnetic media. All reports and documentation required, including chromatograms and mass spectra, must be sequentially paginated and clearly labeled with the laboratory name, EPA Contract number, episode number, and associated EPA sample numbers. Any diskettes, or other electronic media submitted must be similarly labeled. Unless otherwise approved, the laboratories will use the hardcopy reporting forms and automated data reporting formats specified in the megalab contract when reporting data. Finally, each of the laboratories will adhere to a comprehensive data management plan that is consistent with the principles set forth in *Good Automated Laboratory Practices*, EPA Office of Administration and Resources Management, Draft, December 28, 1990. This data management plan must be in place and in use at all times during performance of the megalab contracts.

17.3 SCC Data Management

Data management procedures employed by SCC will include the use of 1) an automated scheduling and tracking system to effectively manage data review and database development activities, 2) standardized data review guidelines to promote consistency in data quality audits across reviewers and over time, 3) a multi-stage data review process designed to maximize the amount of useable data generated in each study, and 4) a standardized data development process to facilitates rapid development of an analytical database with at least 99.9% accuracy.

The automated sample scheduling and tracking system will facilitate the development of up -to-date information concerning work in progress, projected delivery dates, and notice of any problems encountered with laboratory analyses or data turnaround times. To ensure that this information is as complete and accurate as possible, entries will be made into the tracking system at each stage of the sample-to-data sequence. Figure 3 shows the relationship of the computer tracking system to each stage of the data handling process

Figure 3: Relationship of data tracking system to each stage of data production and potential outputs of the sytem



Standardized data review guidelines will be used in this study to facilitate rapid, consistent, accurate, and thorough data quality audits. Data review guidelines already have been developed and are in use for a variety of analyses performed under the "megalab" and other EPA EAD contracts. These guidelines detail method-specific data review procedures for commonly used methods and more general procedures that can be applied to less frequently used methods. Where appropriate, SCC will modify existing data review guidelines as necessary to reflect the methods, method modifications, and data quality objectives employed in the Cook Inlet Contaminant Study.

Although each guideline will be written for a specific method, technique, or group of analytes, all guidelines will specify a general five-stage review process that will ensure data are in proper format, are complete, are contractually compliant, and are usable. SCC chemists will use this multi-stage process to verify the quality of each laboratory submission under the Cook Inlet Contaminant Study. If an error is detected in any stage of the review, SCC staff will initiate corrective action procedures to obtain the maximum amount of usable data from the study. These actions may serve to obtain missing data, correct typographical or transcription errors on data reporting forms, or initiate reanalysis of field or QC samples that do not meet the study DQOs. Additional information regarding this multi-stage data review process is described in Section 20 ("Data Validation and Usability") of this QAPP.

Concurrent with the performance of data quality audits, SCC staff will begin developing a database of analytical results. This database will be formatted in a manner that is consistent with EAD effluent guidelines databases. As with the data quality audits, a multi-stage process of inspections and corrective actions will be used to facilitate timely, efficient construction of databases that are least 99.9% accurate. The database development process will begin with a completeness check to verify that the laboratory has submitted a diskette and that the diskette contains the data in an appropriate format. If deficiencies are found, appropriate corrective action measures will be initiated. After problems have been identified and resolved, the SCC Database Administrator will prepare a "QC Check Report" that displays the results submitted by the laboratory. The SCC chemist responsible for performing the data quality audit will review this QC Check Report to verify that the electronic data accurately reflect the hardcopy submission. Accuracy will be confirmed by spot checking at least 10% of all results that were downloaded directly from an analytical instrument in the laboratory and by performing a 100% QC check of data that were manually entered by the laboratory or SCC. Corrective actions will be taken as needed to resolve deficiencies. Following completion of the data quality audit, the SCC chemist and the Database Administrator will modify the database to reflect data usability determinations. A report, generated to reflect the modified database, will then be reviewed by the SCC chemist to verify database accuracy before submission to EPA.

18.0 Assessments and Response Actions

Several types of assessment activities and corresponding response actions have been identified to ensure that data gathering activities in the Cook Inlet Contaminant Study are conducted as prescribed and to ensure that the measurement quality objectives established in this QAPP and the data quality objectives established by EPA are met. These activities are summarized in Table 4 and discussed in greater detail in Sections 18.1 - 18.7 below.

Table 4 - Assessment and Response Actions

Assessment Measure	Definition	Frequency	Responsible Party	Rationale
Surveillance	Continual or frequent monitoring and verification of the status of an entity and the analysis of records to ensure that specified requirements are being fulfilled	Throughout sample preparation, laboratory analysis, and data review procedures	SCC Sample Sched. & Study Coordinators	Identify and correct analytical problems as soon as they occur to minimize delays; and to notify data users of potential delays as early as possible
Peer Review	A documented critical review of work. Conducted by qualified individuals who are independent, but technically equivalent of those who performed the work.	Performed on 10% of data audits conducted by SCC data reviewers and 100% of data review narratives prepared by SCC data reviewers	SCC data reviewers not responsible for original data review	Ensure that activities are technically adequate, competently performed, properly documented, and satisfy established technical and quality requirements.
Management Systems Review	Qualitative assessment of a data collection operation and/or organization to establish whether the prevailing quality management structure, policies, practices, and procedures are adequate for ensuring that the type and quality of data needed are obtained.	Not scheduled during this study	Not applicable	Standard EAD/SCC procedures will be used for gathering laboratory data gathering; in this study. Such procedures already have been approved following MSRs conducted by GAO and by EPA ORD.
Readiness Review	A systematic documented review of the readiness for the start-up or continued use of a facility, process or activity. Typically conducted before proceeding beyond project milestones and prior to initiation of a major phase of work.	Prior to each laboratory's analysis of field samples collected during the study.	Lab staff and SCC data reviewers	Verify that laboratory is capable of producing precise and accurate results with the method(s) they will use during the study
Technical Systems Audit	A thorough, systematic, on-site, qualitative audit of facilities, equipment, personnel, training, procedures, recordkeeping, data validation, data management, and reporting aspects of a system.	Not required for this study unless specific concerns are raised through discussions with laboratory staff or during other data assessment activities.	If TSAs are deemed necessary, they will be performed by EPA Project Manager and/or EPA QAO, and SCC staff	Ability of each laboratory to adequately analyze and report data will be assessed prior to analysis and continually throughout analyses via other QA/QC measures described in this QAPP. Also, TSAs were conducted at each prime laboratory contractor in the last year
Audit of Data Quality	Systematic and independent examination to determine if quality activities and related results comply with planned arrangements and whether these arrangements are implemented effectively and are suitable to achieve objectives.	100% of laboratory data packages submitted	SCC Data Reviewers	To verify that all data collected meet MQOs established for this study.
Data Quality Assessment	Statistical and scientific evaluation of the data set to determine the validity and performance of the data collection design and statistical test, and to determine the adequacy of the data set for its intended use.	Upon completion of data review and database development	SCC Staff and EPA Data Users	Identify data outliers and evaluate usability of data that failed to meet MQOs

18.1 Surveillance

An SCC Sample Scheduling Coordinator will been assigned to facilitate sample scheduling and to track the location of samples and data throughout the study. During sample collection, the Sample Scheduling Coordinator will maintain communication with field sampling personnel to identify and notify the recipient Sample Prep Laboratory of any delays or anticipated changes to the sampling plan. In the event that these delays or changes impact the laboratory's contract or EPA schedules, the Sample Scheduling Coordinator will notify the EPA Analytical Project Manager and work with EPA, the sampling teams, and other SCC staff to identify and implement an appropriate solution.

When samples are shipped to the Sample Prep Laboratory or the analytical laboratories, the Sample Scheduling Coordinator will contact designated laboratory staff to notify them of the forthcoming shipment(s) and request that they contact SCC if the shipments do not arrive intact as scheduled. Within 24 hours of scheduled sample receipt, the Sample Scheduling Coordinator will contact the laboratory to verify that the samples arrived in good condition, and if problems are noted, will work with the laboratory, the sampling team, and EPA to resolve the problem as quickly as possible to minimize data integrity problems.

The Sample Scheduling Coordinator also will communicate periodically with laboratory staff to monitor the progress of sample preparation, analysis, and data reporting. If technical problems are encountered during sample preparation and analysis, the Sample Scheduling Coordinator will discuss the situation with SCC Study Coordinator. If warranted, the Study Coordinator will identify a technical expert within SCC to assist in resolving the problem, work with the Scheduling Coordinator, the technical expert, laboratory staff, and EPA to identify and implement a solution to the problem. If laboratories fail to deliver data on time, or if the laboratories notify SCC of anticipated reporting delays, the Sample Scheduling Coordinator will notify the Study Coordinator and the EPA Analytical Project Manager of the situation. To the extent possible, the Study Coordinator will adjust schedules and shift resources within SCC as necessary to minimize the impact of laboratory delays on EPA schedules. The Study Coordinator also will immediately notify the Analytical Project Manager of any laboratory delays that are anticipated to impact EPA schedules.

Finally, the SCC Study Coordinator will monitor the progress of the data quality audits (data reviews) and database development to ensure that each laboratory data submission is reviewed in a timely manner. In the event that dedicated staff are not able to meet EPA schedules, the SCC Study Coordinator will work with the SCC Project Manager to identify additional resources who are qualified and capable of reviewing the data in a timely manner. If such resources cannot be identified, and if training new employees is not feasible, the SCC Project Manager will meet with the EPA Analytical Project Manager to discuss an appropriate solution.

18.2 Peer Review

All laboratory results and calculations will be reviewed by the laboratory manager prior to data submission. Any errors identified during this peer review will be returned to the analyst for correction prior to submission of the data package. Following correction of the errors, the Laboratory Manager will verify that the final package is complete and compliant with the contract, and will sign each data submission to certify that s/he has reviewed the package and determined it to be in compliance with the terms and conditions of the contract.

Peer reviews also will be performed within SCC to verify that the data quality audits are being performed consistently over time and across peer reviewers, that the audit findings are technically correct, and that the audits are being performed in accordance with this QAPP. These peer reviews of the SCC data quality audit process will be performed on at least 5% of the data packages received in this study. Peer reviewers will be charged with evaluating the completeness of the original data review, the technical accuracy of the reviewers findings, and the technical accuracy of the analytical database developed to store results associated with the data package. The SCC Study Coordinator will be responsible for identifying and assigning qualified peer reviewers and for selecting packages to be peer reviewed. Qualified peer reviewers will include any staff members that have been trained in SCC data review procedures, that are experienced in reviewing data similar to those being reviewed, and are familiar with the requirements of the Cook Inlet Contaminant Study and this QAPP. Data packages will be selected for peer review in such a way as to maximize the number of data reviewers and types of data (e.g., pesticides, dioxins, metals, etc) that are subjected to this peer review process. To the extent possible, these peer reviews will be performed after the primary data reviewer has drafted a written narrative describing the results of his/her audit, but before this narrative is submitted to EPA.

To ensure that the findings of each data quality audit are documented in a consistent and technically accurate manner, SCC staff will peer review 100% of the data review summaries (narratives) prepared for this study. Each data review summary will be subjected to at least two levels of peer review, and each peer reviewer will be charged with evaluating the clarity, technical accuracy, and the grammatical quality of the data review summary.

18.3 Management Systems Review

All data gathering procedures routinely employed by EAD's Analytical Methods Staff (AMS) will be applied to the data gathering activities associated with this study. These standard AMS procedures have been subjected to periodic Management Systems Reviews by external sources interested in assessing the overall quality of EAD's data collection operation. In every case, the external reviewers found AMS' data gathering process to be technically sound.

Because the data gathered in the Cook Inlet Contaminant Study will be collected in accordance with standard AMS protocols, and because these protocols have repeatedly withstood external reviews, EPA has concluded that a formal Management Systems Review focused on the Cook Inlet Contaminant Study is unnecessary.

18.4 Readiness Review

A readiness review of each laboratory's capability to produce precise and accurate results with the methods specified in this study will be performed before the laboratories are allowed to analyze field samples collected during the study. As part of the readiness reviews, the laboratories will submit data demonstrating that they are capable of analyzing a known, reference matrix with the methods to be used in this study. In most cases, laboratories will meet this requirement by performing IPR tests. IPR tests consist of preparing four replicate aliquots that contain the target pollutants, analyzing these replicate aliquots with the specified method, and calculating the average percent recovery and standard deviation of the measured aliquots. If the average percent recovery and standard deviation meet pre-defined acceptance criteria, the laboratory is considered to be qualified, or ready, to perform the analyses.

On a case by case basis, EPA and SCC may decide to accept alternate data in lieu of IPR data for the readiness reviews. In such cases, the alternate data must provide as much information about laboratory readiness as would the IPR samples. Examples of acceptable non-IPR data that might be used for a readiness review include: performance evaluation (PE) sample data; ongoing QC data gathered over a period of time, or MDL study data.

Readiness reviews will be performed by SCC data reviewers, who will document and forward their findings to the SCC Study Coordinator. If problems are identified during these reviews, the SCC Data Reviewer(s) and Study Coordinator will work with the laboratory, to the extent possible, to resolve the problem. If the problem cannot be resolved within the time frame required by EPA or within the scope of the laboratory's existing contract, the SCC Study Coordinator will notify the EPA Analytical Project Manager immediately.

18.5 Technical Systems Audit

Under the terms of their contracts with EPA, each laboratory participating in this study must be prepared for and willing to undergo an on-site, or technical systems, audit of its facilities, equipment, staff, and sample analysis, training, recordkeeping, data validation, data management, and data reporting procedures. EPA will conduct such audits for individual laboratories participating in this study only if the results of the readiness reviews, data quality audits, and surveillance suggest serious or chronic laboratory problems that warrant on-site examinations and discussion with laboratory personnel. If such an audit is determined to be necessary, a standardized audit checklist will be used to facilitate an audit walkthrough and document audit findings. Audit participants will include the EPA Analytical Project Manager or the EPA Quality Assurance Manager (or a qualified EPA staff member designated by the EPA QAM) and an SCC staff member experienced in conducting laboratory audits. The EPA staff member will be responsible for leading the audit and conducting a post-audit debriefing to convey significant findings to laboratory staff at the conclusion of the audit. The SCC staff member will be responsible for gathering pre-audit documentation of problems that necessitated the audit, customizing the audit checklist as necessary to ensure that those problems are addressed during the audit, documenting audit findings on the audit checklist during the audit, and drafting a formal report of audit findings for review by EPA.

18.6 Data Quality Audits

Every laboratory data package submitted under this study will be subjected to a data quality audit. These data quality audits will be performed by qualified SCC data review staff who have been trained in procedures for performing data quality audits and who are familiar with the laboratory methods used to prepare the data packages. These data quality audits will be performed using a multi-stage review process designed to identify and correct data deficiencies as early as possible in order to maximize the amount of usable data generated during this study. Section 20 of this QAPP describes the data quality audit process in greater detail.

18.7 Data Quality Assessment

Upon completion of each data quality audit, the SCC Data Reviewer will work with SCC's database development staff to create an analytical database that contains all field sample results from the Cook Inlet Contaminant Study. A separate database containing results of QC samples generated during the study also will be prepared. Details regarding database construction are provided below in Section 19.

At selected intervals and upon completion of the study, SCC's database development staff will perform statistical analyses to verify the accuracy of the database. The statistical procedures will be directed at evaluating the overall quality of the database against data quality objectives established for the study, and in identifying trends in field and QC results obtained during the study. SCC staff will document their findings and recommendations concerning this data quality assessment in a written report to EPA.

19.0 Reports to Management

Following completion of each data quality audit and assessment, SCC chemists will prepare and submit written reports, in narrative format, that describe data quality limitations and SCC recommendations concerning data use.

In order to facilitate sample and data tracking during the course of the Cook Inlet Contaminant Study, SCC also will prepare a monthly status report that provides a complete listing of the Episodes scheduled, the analyses required under each Episode, the laboratory associated with each analysis type, the date of sample receipt at the laboratory, the date of data receipt at SCC, the SCC data review completion date, and the SCC database completion date. This report will enable the Analytical Project Manager to quickly determine the status of individual Cook Inlet Contaminant Study components. This monthly status reports also will be used by the EPA Analytical Project Manager and other EPA staff to facilitate decisions regarding the study and to inform senior EPA managers of the project status.

Upon request, SCC also will be required to provide a weekly report that describes the status of all current sampling, analysis, and data review activities, and periodic database status reports that provide up-to-date information concerning database developments that occurred since distribution of previous reports.

20.0 Data Review, Validation, and Verification

A multi-stage data review process, as summarized in Section 17 and detailed below, will be used to evaluate the quality of all data submitted in the Cook Inlet Contaminant Study. Acceptance criteria against which data will be evaluated will include 1) DQOs and MQOs detailed in this QAPP, 2) applicable QC acceptance criteria outlined in the methods, and 3) best professional judgement (BPJ) of SCC chemists responsible for performing data quality assessments.

In the first stage of the data review process, SCC chemists will perform a "Data Completeness Check" in which all elements in each laboratory submission will be evaluated to verify that results for all specified samples are provided, that data are reported in the correct format, and that all relevant information, such as preparation and analysis logs, are included in the data package. Corrective action procedures will be initiated if deficiencies are noted.

The second stage of the data review process will focus on an "Instrument Performance Check" in which the SCC chemists will verify that calibrations, calibration verifications, standards, and calibration blanks were analyzed at the appropriate frequency and met method or study performance specifications. If errors are noted at this stage, corrective action procedures will be initiated immediately.

Stage three of the data review process will focus on a "Laboratory Performance Check" in which SCC staff will verify that the laboratory correctly performed the required analytical procedures and was able to demonstrate a high level of precision and accuracy. This stage includes evaluation of QC elements such as the IPR and OPR tests, preparation and laboratory blanks, and reference standards. Corrective action procedures will be initiated with the laboratories to resolve any deficiencies identified.

In stage four of the data review process, the SCC chemist will perform a "Method/Matrix Performance Check" to discern whether any QC failures are a result of laboratory performance or difficulties with the method or sample matrix. Data evaluated in this stage will include matrix spike, matrix spike duplicate, duplicate sample, labeled compound, and surrogate spike results. The SCC chemist also will verify that proper sample dilutions were performed and that necessary sample cleanup steps were taken. If problems are encountered, the SCC chemist will immediately implement corrective actions.

Finally, SCC will perform a "Data Quality and Usability Assessment" in which the overall quality of data is evaluated against the DQOs and MQOs detailed in this QAPP. Results of this assessment will be documented in written data review narratives that SCC will submit to EPA. To expedite the process, these narratives will follow a standardized format and, wherever possible, utilize standardized language to communicate data limitations and SCC recommendations concerning data quality.

The EPA Study Manager will utilize the SCC-generated data review narratives to make final data usability determinations regarding each set of sample results for performing risk assessment activities.

References

(1) Combined Workplan and Quality Assurance Project Plan for the Cook Inlet Contaminant Study, Prepared for EPA by Arthur D. Little, Inc., June 5, 1997; Reference 33648

(2) *Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels*, July 1996; EPA 821-R-96-008. Draft.

(3) *Method 1631: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry*, July 1996; EPA 821-R-96-012. Draft.

(4) *NOAA Technical Memorandum NOS ORCA 71*: "Sampling and Analytical Methods of the National Status and Trends Program National Benthic Surveillance and Mussel Watch Projects 1984 - 1992"; Volume III, Comprehensive Descriptions of Elemental Analytical Methods; G.C. Lauenstein and A.Y. Cantillo,editors; July 1993; Silver Spring, MD.

(5) Bloom, N.S. 1989. "Determination of Picogram Levels of Methyl Mercury by Aqueous Phase Ethylation, Followed by Cryogenic Gas Chromatography with Cold Vapor Atomic Fluorescence Detection." Can. J. Fish Aq. Sciene, 46: 1131-1140.

(6) Crecelius, E.A., N.S. Bloom, C.E. Cowan, and E.a. Jenne. 1986. "Speciation of Selenium and Arsenic in Natural Waters and Sediments, Volume 2: Arsenic Speciation." Final Report prepared for Electric Power Research Institute, Palo Alto, California by Batelle Pacific Northwest Laboratories, Richland, Washington.

(7) *Guide to Method Flexibility and approval of EPA Water Methods*, EPA Office of Water, Engineering and Analsyis Division (4303), Washington DC 20460. EPA-821-D-96-004. December 1996.

Appendix A

List of Target Organohalide Pesticides

Appendix A - List of Target Organohalide Pesticides and PCBs

Chlordane (total)
cis-Chlordane
trans-Chlordane
cis-Nonachlor
trans-Nonachlor
Oxychlordane
DDT (total)
4,4'-DDT
2,4'-DDT
4,4'-DDD
2,4'-DDD
4,4'-DDE
2,4'-DDE

Dieldrin
Endosulfan (total)
Endosulfan I
Endosulfan II
Endrin
Heptachlor epoxide
Hexachlorobenzene
Lindane
Mirex
Pentachloroanisole
Toxaphene

PCB-1016
PCB-1221
PCB-1232
PCB-1242
PCB-1248
PCB-1254
PCB-1260